

## Supporting Information

# Base-Promoted Transition-metal-free Phosphorylation/Acylation of 2-Vinyl Pyridines with Ph<sub>2</sub>PCl and Carboxylic Acids.

Zhichao Li,<sup>a</sup> Lianze Zong,<sup>a</sup> Xixi Hu,<sup>a</sup> Xinjin Li,<sup>a</sup> Fenggang Sun,<sup>a</sup> Yunhui Dong,<sup>a</sup> Zhengbao Xu,<sup>b</sup> Hui  
Liu, \*<sup>a</sup> and Lizhi Zhang\*<sup>a</sup>

<sup>a</sup> School of Chemistry and Chemical Engineering, Shandong University of Technology, Zibo,  
255000, Shandong, P. R. of China.

<sup>b</sup> School of Life Sciences and Medicine, Shandong University of Technology, Zibo, 255000,  
Shandong, P. R. of China.

E-mail: lizhizhangsdut@foxmail.com; zhanglizhi@sdut.edu.cn

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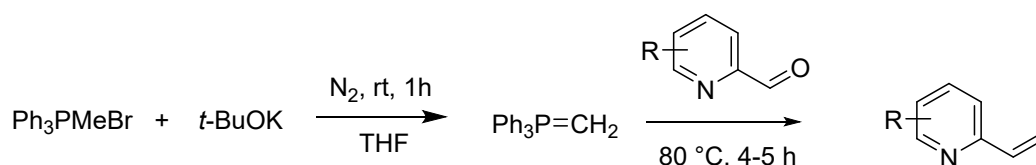
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## 1. General Information

$^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{31}\text{P}$  and  $^{19}\text{F}$  NMR spectra were recorded in  $\text{CDCl}_3$  on 400 MHz spectrometers. The chemical shifts of  $^1\text{H}$  NMR spectra in  $\text{CDCl}_3$  were determined with TMS as the internal standard ( $\delta = 0.0$  ppm). The chemical shifts in  $^{13}\text{C}$  NMR spectra were determined based on the chemical shift of  $\text{CDCl}_3$  ( $\delta = 77.0$  ppm). Multiplicities are given as s (singlet), d (doublet), t (triplet), dd (doublet of doublet), td (triplet of doublet) or m (multiplet). Deuterated solvents were purchased from Cambridge Isotope Laboratories. HRMS spectra were measured using a Q-TOF instrument equipped with an ESI source. The X-ray single crystal analysis was carried on a XtaLAB Synergy Single crystal diffractometer.

Unless otherwise noted, the chemicals are either commercially available or known compounds that can be prepared following reported procedures. Synthesis of 2-vinyl pyridines utilizing the protocol reported in the literature [1]. All the solvents are anhydrous or of analytical grade, and were used without further purification. Analytical TLC was performed with silica gel GF254 plates, and 200–300 mesh silica gel was employed for column chromatography.

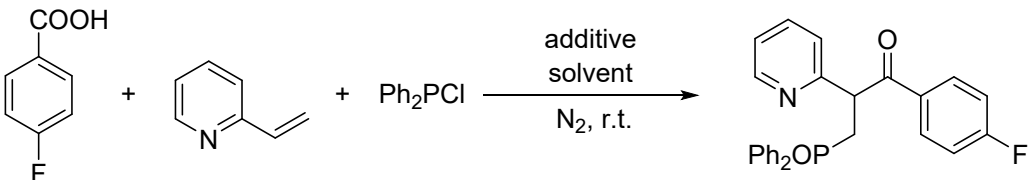
## 2. The Synthesis of Starting Materials [1]



To a solution of methyltriphenylphosphonium bromide (2 equiv) in THF, *t*-BuOK (3 equiv) was added in three times. After stirring the reaction mixture at room temperature for 1 hour under nitrogen atmosphere. Then, the corresponding aldehyde (1 equiv) was added portion wise and the reaction was stirred reflux at 80 °C for 4-5 hours. Then the reaction mixture was quenched by addition of water. The organic layer is separated and the aqueous layer is extracted with DCM (3 \*10 mL). The combined organic phases are dried over  $\text{Na}_2\text{SO}_4$  and evaporated under reduced pressure. Purification of the residue by flash column chromatography on silica gel (PE/EA solvent mixtures as eluent, 1%  $\text{NEt}_3$  was added) affords the desired product.

### 3. Modification of the typical reaction conditions

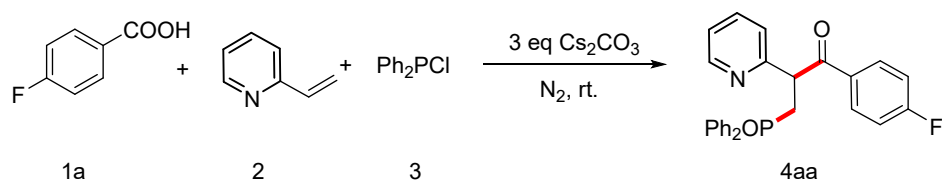
Table S1 Modification of the typical reaction conditions.



Entry	Solvent (2 ml)	Base	Ph <sub>2</sub> PCl (eq)	Time	Yield <sup>b</sup> (%)
1	DMF	3 eq Cs <sub>2</sub> CO <sub>3</sub>	3 eq	16 h	ND <sup>c</sup>
2 <sup>b</sup>	Toluene	3 eq Cs <sub>2</sub> CO <sub>3</sub>	3 eq	16 h	8 %
3 <sup>c</sup>	DMSO	3 eq Cs <sub>2</sub> CO <sub>3</sub>	3 eq	16 h	ND <sup>c</sup>
4	CH <sub>3</sub> CN	3 eq Cs <sub>2</sub> CO <sub>3</sub>	3 eq	16 h	14 %
5 <sup>c</sup>	THF	3 eq Cs <sub>2</sub> CO <sub>3</sub>	3 eq	16 h	5 %
<b>6</b>	<b>DCM</b>	<b>3 eq Cs<sub>2</sub>CO<sub>3</sub></b>	3 eq	<b>16 h</b>	<b>86 %</b>
7	DCM	1.5 eq Cs <sub>2</sub> CO <sub>3</sub>	1.5 eq	16 h	70 %
8	DCM	5 eq Cs <sub>2</sub> CO <sub>3</sub>	5 eq	16 h	42 %
9	DCM	none	3 eq	16 h	66 %
10	DCM	3 eq DMAP	3 eq	16 h	ND <sup>c</sup>
11	DCM	3 eq NaH	3 eq	16 h	43 %
12	DCM	3 eq KOH	3 eq	16 h	23 %
13	DCM	3 eq K <sub>2</sub> CO <sub>3</sub>	3 eq	16 h	37 %
14	DCM	3 eq Na <sub>2</sub> CO <sub>3</sub>	3 eq	16 h	25 %
15	DCM	3 eq NaOH	3 eq	16 h	36 %
16	DCM	3 eq DBU	3 eq	16 h	9 %
17	DCM	3 eq Et <sub>3</sub> N	3 eq	16 h	44 %
18	DCM	3 eq Cs <sub>2</sub> CO <sub>3</sub>	3 eq	8 h	61%
19	DCM	3 eq Cs <sub>2</sub> CO <sub>3</sub>	3 eq	12 h	70%
18	DCM (1 mL)	3 eq Cs <sub>2</sub> CO <sub>3</sub>	3 eq	16 h	49 %
19	DCM (5 mL)	3 eq Cs <sub>2</sub> CO <sub>3</sub>	3 eq	16 h	80 %
20	DCM (10 mL)	3 eq Cs <sub>2</sub> CO <sub>3</sub>	3 eq	16 h	78 %

<sup>a</sup> Reaction conditions: 4-fluorobenzoic acid (0.2 mmol, 1 eq), 2-vinylpyridine (0.4 mmol, 2 eq), chlorodiphenyl phosphine (0.6 mmol, 3 eq), solvent (2 mL), 1 atm N<sub>2</sub>. <sup>b</sup> Isolated yield. <sup>c</sup> ND means none detected.

### 4. Typical reaction conditions.



**Reaction condition A:**

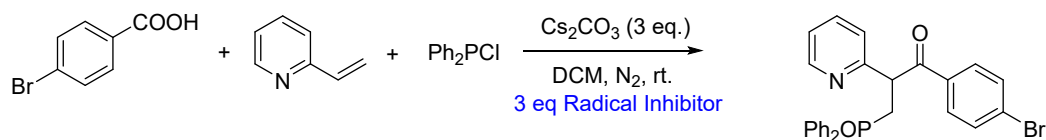
A mixture of **1a** (1 eq, 0.2 mmol) and Cs<sub>2</sub>CO<sub>3</sub> (3 eq, 0.6 mmol) were dissolved in DCM (2 mL), then the reaction tube was purged with N<sub>2</sub> for three times. The **2** (2 eq, 0.4 mmol) and **3** (3 eq, 0.6 mmol) was added into the tube with a micro injector. The reaction mixture was stirred at room temperature for 16 hours, then neutralized with Na<sub>2</sub>CO<sub>3</sub> solution to pH =7 ~ 8. The two-phase was separated, and the aqueous phase was extracted with DCM (3\*5 mL), the organic phase was washed with brine for one time, dried over Na<sub>2</sub>SO<sub>4</sub>. After filtered, the solvent was removed under reduced pressure, and purified by flash chromatography on silica gel to afford **4aa** as white solid.

#### Reaction condition B:

A mixture of **1a** (1 eq, 0.5 mmol) and Cs<sub>2</sub>CO<sub>3</sub> (3 eq, 1.5 mmol) were dissolved in DCM (5 mL), then the reaction tube was purged with N<sub>2</sub> for three times. The **2** (2 eq, 1.0 mmol) and **3** (3 eq, 1.5 mmol,) was added into the tube with a micro injector. The reaction mixture was stirred at room temperature for 16 hours then neutralized with Na<sub>2</sub>CO<sub>3</sub> solution to pH =7 ~ 8. The two-phase was separated, and the aqueous phase was extracted with DCM (3\*10 ml), the organic phase was washed with brine for one time, dried over Na<sub>2</sub>SO<sub>4</sub>. After filtered, the solvent was removed under reduced pressure, and purified by flash chromatography on silica gel to afford **4aa** as white solid.

## 5. Mechanism Verification Experiment.

### Control Reactions with Radical Inhibitor

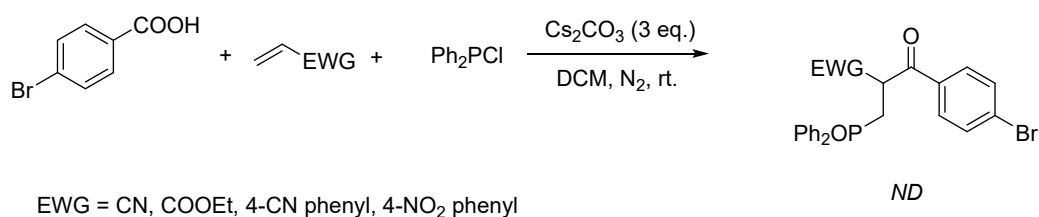


Radical Inhibitor	Yield
TEMPO	42.4%
BHT	87%

A mixture of 4-bromobenzoic acid (1 eq, 0.2 mmol), radical inhibitor (3 eq, 0.6 mmol) and Cs<sub>2</sub>CO<sub>3</sub> (3 eq, 0.6 mmol) were dissolved in DCM (2 mL), then the reaction tube was purged with N<sub>2</sub> for three times. 2-vinylpyridine (2 eq, 0.4

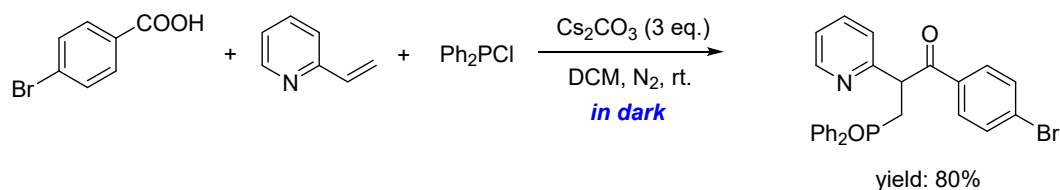
mmol) and chlorodiphenyl phosphine (3 eq, 0.6 mmol) was added into the tube with a micro injector. The reaction mixture was stirred at room temperature for 16 hours, then neutralized with Na<sub>2</sub>CO<sub>3</sub> solution to pH =7 ~ 8. The two-phase was separated, and the aqueous phase was extracted with DCM (3\*5 ml), the organic phase was washed with brine for one time, dried over Na<sub>2</sub>SO<sub>4</sub>. After filtered, the solvent was removed under reduced pressure, and purified by flash chromatography on silica gel to afford the desired product.

### Control Reactions with Electron-deficient Alkenes



A mixture of 4-bromobenzoic acid (1 eq, 0.2 mmol) and Cs<sub>2</sub>CO<sub>3</sub> (3 eq, 0.6 mmol) were dissolved in DCM (2 ml), then the reaction tube was purged with N<sub>2</sub> for three times. The alkenes (2 eq, 0.4 mmol) and chlorodiphenyl phosphine (3 eq, 0.6 mmol) was added into the tube with a micro injector. The reaction mixture was stirred at room temperature for 16 hours. No target products were detected through TLC analysis and GC-MS.

### The Reaction Conducted in Dark



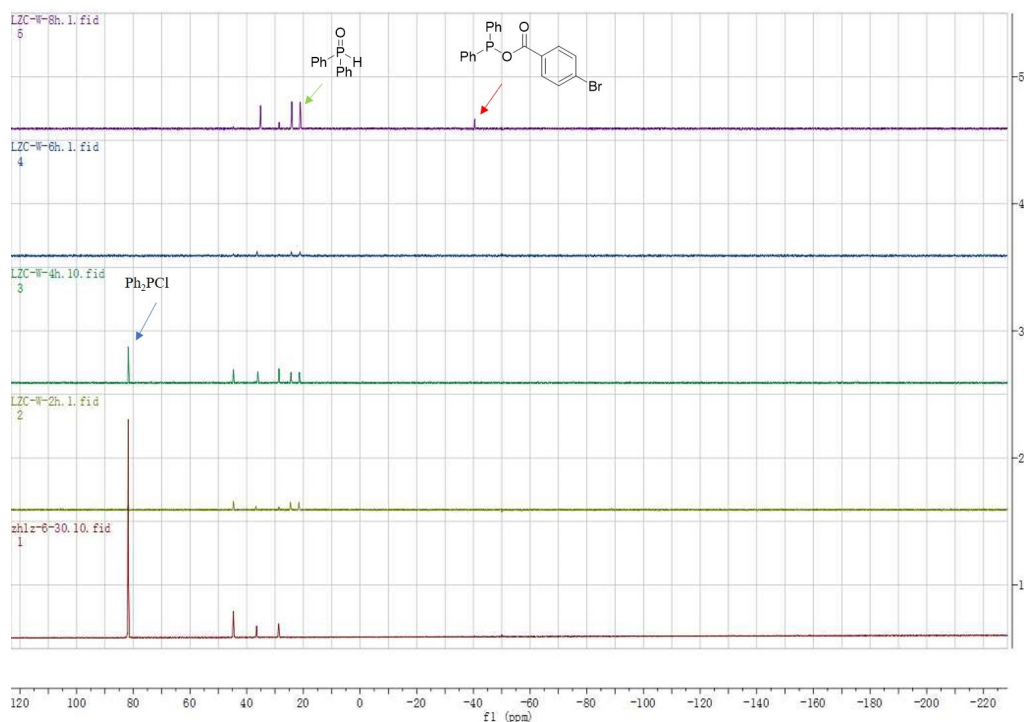
A mixture of 4-bromobenzoic acid (1 eq, 0.2 mmol) and Cs<sub>2</sub>CO<sub>3</sub> (3 eq, 0.6 mmol) were dissolved in DCM (2 mL), then the reaction tube was purged with N<sub>2</sub> for three times. The alkenes (2 eq, 0.4 mmol) and chlorodiphenyl phosphine (3 eq, 0.6 mmol) was added into the tube with a micro injector. Then the reaction tube was wrapped with tin foil. The reaction mixture was stirred at room temperature for 16 hours then neutralized with Na<sub>2</sub>CO<sub>3</sub> solution to pH =7 ~ 8. The two-phase was separated, and the aqueous phase was extracted with DCM (3\*5 ml), the organic phase was washed with brine for one time, dried

over  $\text{Na}_2\text{SO}_4$ . After filtered, the solvent was removed under reduced pressure, and purified by flash chromatography on silica gel to afford **4ac** in a yield of 80% (78 mg).



### NMR detection experiments <sup>[3]</sup>

A mixture of 4-bromobenzoic acid (1 eq, 0.2 mmol) and chlorodiphenyl phosphine (3 eq, 0.6 mmol) were dissolved in  $\text{CDCl}_3$  (2 mL), then the reaction tube was purged with  $\text{N}_2$  for three times. Samples (100  $\mu\text{L}$ ) were taken via a syringe in every two hours. The sample was diluted by  $\text{CDCl}_3$  and detected with  $^{31}\text{P}$  NMR.

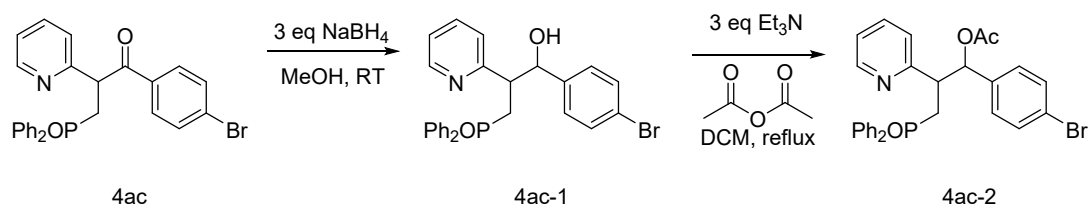


## 6. Gram Scale Preparation of 4ac.

A mixture of 4-bromobenzoic acid (1 eq, 35 mmol) and  $\text{Cs}_2\text{CO}_3$  (3 eq, 105 mmol) were dissolved in DCM (150 mL), then the reaction tube was purged with  $\text{N}_2$  for three times. The 2-vinylpyridine (2 eq, 70 mmol) and chlorodiphenyl phosphine (3 eq, 105 mmol) was added into the flask with syringe. The reaction mixture was stirred at room temperature for 10 hours. Then neutralized with  $\text{Na}_2\text{CO}_3$  solution to  $\text{pH} = 7 \sim 8$ . Then the two-phase was separated, and the aqueous phase was extracted with DCM (3\*100 mL), the organic phase was washed with brine for one time, dried over  $\text{Na}_2\text{SO}_4$ . After filtered, the solvent was removed under reduced pressure. The product was recrystallized in DCM and EA to give **4ac** in a yield of 86% (14.75 g).



## 7. Transformation of The Final Product.



Compound **4ac** (1 eq, 0.25 mmol) was dissolved in MeOH (5 mL), then  $\text{NaBH}_4$  (3 eq, 0.75 mmol) was added slowly. Then the mixture was stirred at RT for 4h. After the reaction was completed (TLC analysis), Saturated  $\text{NH}_4\text{Cl}$  (20 ml) was added, the suspension was extracted with EtOAc (3\*30 mL), the combined organic phase was washed with brine for one time and dried over  $\text{Na}_2\text{SO}_4$ , concentrated under reduced pressure, purified by flash chromatography on silica gel to afford **4ac-1** as a white solid.

A mixture of **4ac-1** (1 eq, 0.2 mmol) and  $\text{Et}_3\text{N}$  (3 eq, 0.6 mmol) were dissolved in DCM (5 mL), and acetic anhydride (2 mL) was added dropwise. Then the mixture

was refluxing for 10 h. After the reaction was completed (TLC analysis), H<sub>2</sub>O (20 ml) was added, the suspension was extracted with EtOAc (3\*30 mL), the combined organic phase was washed with brine for one time and dried over Na<sub>2</sub>SO<sub>4</sub>, concentrated under reduced pressure, purified by flash chromatography on silica gel to afford **4ac-2** as a white solid.

## 8. X-ray Single Crystal Diffraction Data of **4ac**.

Sample preparation: A solution of compound **4ac** (15 mg) in DCM (1mL) was placed in a vial (5 mL). EA (1 mL) was added to the solution with a dropper, then the vial was placed in refrigerator. The single crystal **4ac** was obtained by slowly evaporating the mixed solvent at -5°C. Single crystal measurement: the structure of **4ac** was determined at room temperature (292-293 K) using an XtaLAB Synergy Single crystal diffractometer with a HyPix-6000HE detector.

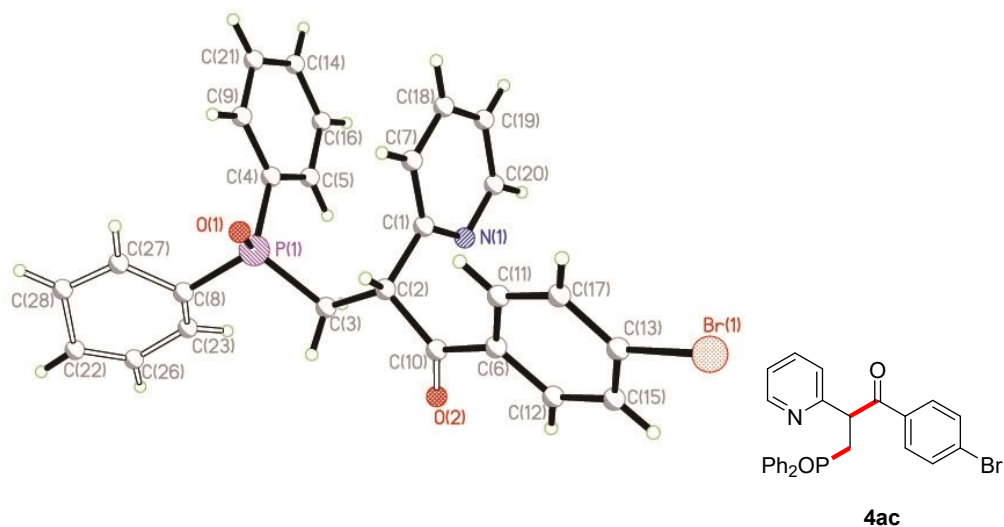


Table S2. Crystal data and structure refinement for **4ac**.

Empirical formula	<i>C</i> 26 <i>H</i> 21.30 <i>Br</i> <i>N</i> <i>O</i> 2 <i>P</i>
Formula weight	490.62
Temperature	300.60(10) <i>K</i>
Wavelength	1.54184 <i>Å</i>
Crystal system, space group	Triclinic, <i>P</i> -1
Unit cell dimensions	<i>a</i> = 5.7965(3) <i>Å</i> <i>alpha</i> = 95.046(3) deg.



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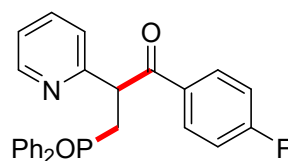
	$b = 8.8198(3) \text{ \AA}$	$\beta = 92.380(3) \text{ deg.}$
	$c = 23.4827(7) \text{ \AA}$	$\gamma = 107.933(4) \text{ deg.}$
Volume	$1134.80(8) \text{ \AA}^3$	
Z, Calculated density	2, $1.436 \text{ Mg/m}^3$	
Absorption coefficient	$3.316 \text{ mm}^{-1}$	
F(000)	501	
Crystal size	$0.08 \times 0.06 \times 0.05 \text{ mm}$	
Theta range for data collection	$1.894 \text{ to } 77.591 \text{ deg.}$	
Limiting indices	$-5 \leq h \leq 7, -11 \leq k \leq 11, -29 \leq l \leq 27$	
Reflections collected / unique	12619 / 4466 [ $R(\text{int}) = 0.0545$ ]	
Completeness to theta = 67.684	99.0 %	
Absorption correction	<i>Semi-empirical from equivalents</i>	
Max. and min. transmission	1.00000 and 0.36085	
Refinement method	<i>Full-matrix least-squares on <math>F^2</math></i>	
Data / restraints / parameters	4466 / 61 / 325	
Goodness-of-fit on $F^2$	1.075	
Final R indices [ $I > 2\sigma(I)$ ]	$R1 = 0.0758, wR2 = 0.2177$	
R indices (all data)	$R1 = 0.0847, wR2 = 0.2282$	
Extinction coefficient	n/a	
Largest diff. peak and hole	$0.975 \text{ and } -1.517 \text{ e.\AA}^{-3}$	

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## 9. Physical Data for The Products.

### 1. 3-(diphenylphosphoryl)-1-(4-fluorophenyl)-2-(pyridin-2-yl)propan-1-one(4aa)

A white solid, 74.2 mg, 86 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 %  $\text{Et}_3\text{N}$ )



$^1\text{H NMR}$  (400 MHz, Chloroform-d):  $\delta$  8.33 (s, 1H), 7.94 (t,  $J = 6.0 \text{ Hz}$ , 2H), 7.70 (t,  $J = 8.8 \text{ Hz}$ , 2H), 7.56 (t,  $J = 8.8 \text{ Hz}$ , 2H), 7.42 - 7.33 (m, 5H), 7.28 - 7.23 (m, 3H),

6.97 (t, J = 8.4 Hz, 2H), 6.90 (t, J = 4.4 Hz, 1H), 5.43- 5.38 (m, 1H), 3.41 - 3.30 (m, 1H), 3.10 - 3.02 (m, 1H).

**<sup>13</sup>C NMR (101 MHz, Chloroform-d):** δ 195.34 (d, J = 8.6 Hz), 165.49 (d, J = 256.0 Hz), 157.55 (d, J = 6.2 Hz), 149.74, 136.68, 133.27 (d, J = 100.8 Hz), 132.12 (d, J = 2.9 Hz), 133.27 (d, J = 99.8 Hz), 131.70 (d, J = 2.7 Hz), 131.60 (d, J = 9.4 Hz), 131.26 (d, J = 2.6 Hz), 130.62 (d, J = 9.4 Hz), 130.58 (d, J = 9.6 Hz), 128.43 (d, J = 25.4 Hz), 128.32 (d, J = 25.4 Hz), 123.94, 122.14, 115.49 (d, J = 21.9 Hz), 49.14, 31.85 (d, J = 71.6 Hz).

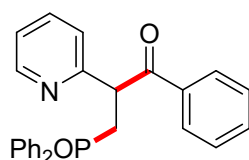
**<sup>31</sup>P NMR (162 MHz, Chloroform-d):** δ 30.29.

**<sup>19</sup>F NMR (376 MHz, Chloroform-d):** δ -105.02.

**HRMS (ESI, m/z):** Calculated for C<sub>26</sub>H<sub>21</sub>FN<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup>430.4312, found 430.3124

## 2. 3-(diphenylphosphoryl)-1-phenyl-2-(pyridin-2-yl)propan-1-one (4ab)

A white solid, 70 mg, 85 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):** δ 8.25 (d, J = 4.4 Hz, 1H), 7.84 (d, J = 7.6 Hz, 2H), 7.64 (t, J = 8.4 Hz, 2H), 7.50 (t, J = 8.0 Hz, 2H), 7.37 - 7.24 (m, 7H), 7.22 - 7.18 (m, 4H), 6.81 (t, J = 6.0 Hz, 1H), 5.43 - 5.37 (m, 1H), 3.35 - 3.27 (m, 1H), 3.08 - 3.00 (m, 1H).

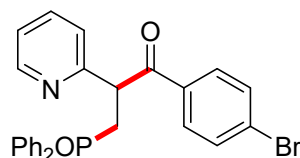
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):** δ 196.83 (d, J = 8.8 Hz), 157.65 (d, J = 5.9 Hz), 149.66, 136.56, 135.81, 133.41 (d, J = 100.1 Hz), 132.42 (d, J = 92.4 Hz), 131.62 (d, J = 2.6 Hz), 131.18 (d, J = 2.8 Hz), 130.64 (d, J = 9.5 Hz), 130.59 (d, J = 9.5 Hz), 128.88, 128.39 (d, J = 28.0 Hz), 128.32, 128.28 (d, J = 28.0 Hz), 124.11, 122.00, 49.15, 31.82 (d, J = 72.0 Hz).

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):** δ 30.42.

**HRMS (ESI, m/z):** Calculated for C<sub>26</sub>H<sub>22</sub>N<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup>412.4408, found 412.2136

## 3. 1-(4-bromophenyl)-3-(diphenylphosphoryl)-2-(pyridin-2-yl)propan-1-one (4ac)

A white solid, 191.5 mg, 78 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N).



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):** δ 8.33 (d, J = 3.2 Hz, 1H), 7.79 (d, J = 8.0 Hz, 2H), 7.72 (t, J = 8.4 Hz, 2H), 7.59 (t, J = 8.4 Hz, 2H), 7.45 - 7.33 (m, 7H), 7.27 (t, J = 8.4 Hz, 3H), 6.90 (t, J = 5.6 Hz, 1H), 5.45-5.39 (m, 1H), 3.46 - 3.38 (m, 1H), 3.12 - 3.04 (m, 1H).

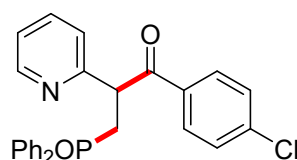
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):**  $\delta$  195.77 (d, J = 8.7 Hz), 157.26 (d, J = 6.1 Hz), 149.64, 136.60, 134.36, 133.19 (d, J = 100.3 Hz), 132.12 (d, J = 99.5 Hz), 131.61 (d, J = 2.8 Hz), 131.54, 131.17 (d, J = 2.7 Hz), 130.49 (d, J = 9.4 Hz), 130.43 (d, J = 9.6 Hz), 130.30, 128.34 (d, J = 26.6 Hz), 128.22 (d, J = 26.5 Hz), 128.03, 123.90, 122.07, 49.13, 32.64 (d, J = 71.9 Hz).

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):**  $\delta$  30.12.

**HRMS (ESI, m/z):** Calculated for C<sub>26</sub>H<sub>21</sub>BrN<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup> 491.3368, found 491.2162.

#### 4. 1-(4-chlorophenyl)-3-(diphenylphosphoryl)-2-(pyridin-2-yl)propan-1-one (4ad)

A white solid, 86.5 mg, 97 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):**  $\delta$  8.36 (d, J = 4.4 Hz, 1H), 7.88 (d, J = 7.6 Hz, 2H), 7.74 (t, J = 8.0 Hz, 2H), 7.60 (t, J = 8.4 Hz, 2H), 7.47 - 7.36 (m, 5H), 7.32 - 7.26 (m, 5H), 6.93 (t, J = 5.6 Hz, 1H), 5.46 - 5.40 (m, 1H), 3.46 - 3.37 (m, 1H), 3.13 - 3.05 (m, 1H).

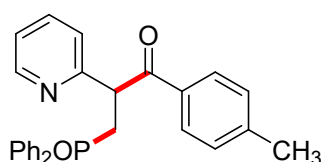
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):**  $\delta$  195.63 (d, J = 8.4 Hz), 157.41 (d, J = 6.3 Hz), 149.63, 139.24, 136.61, 134.09, 133.23 (d, J = 96.4 Hz), 132.24 (d, J = 95.6 Hz), 131.61 (d, J = 2.7 Hz), 131.20 (d, J = 2.7 Hz), 130.54 (d, J = 9.5 Hz), 130.50 (d, J = 9.6 Hz), 130.23, 128.57, 128.36 (d, J = 24.5 Hz), 128.24 (d, J = 24.4 Hz), 123.85, 122.07, 49.17, 31.76 (d, J = 71.8 Hz).

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):**  $\delta$  30.27.

**HRMS (ESI, m/z):** Calculated for C<sub>26</sub>H<sub>21</sub>Cl<sub>1</sub>N<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup> 446.8828, found 446.1029

#### 5. 3-(diphenylphosphoryl)-2-(pyridin-2-yl)-1-(p-tolyl)propan-1-one (4ae)

A yellow solid, 64.6 mg, 76 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):**  $\delta$  8.33 (d, J = 4.4 Hz, 1H), 7.84 (d, J = 7.6 Hz, 2H), 7.75 - 7.69 (m, 2H), 7.58 (t, J = 10.4 Hz, 1H), 7.52 - 7.49 (m, 1H), 7.44 - 7.31 (m, 4H), 7.27 (s, 3H), 7.12 (d, J = 7.6 Hz, 2H), 6.88 (t, J = 6.0 Hz, 1H), 5.50 - 5.44 (m, 1H), 3.42 - 3.34 (m, 1H), 3.17 - 3.09 (m, 1H), 2.31 (s, 3H).

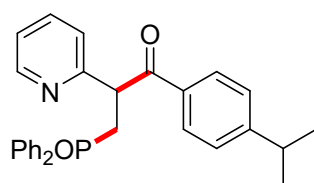
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):** δ 196.33 (d, J = 9.2 Hz), 157.66 (d, J = 5.5 Hz), 149.55, 143.81, 136.64, 133.15, 133.35 (d, J = 100.2 Hz), 132.28 (d, J = 99.2 Hz), 131.66 (d, J = 2.6 Hz), 131.17 (d, J = 2.6 Hz), 130.65 (d, J = 9.3 Hz), 130.56 (d, J = 8.2 Hz), 129.08, 128.86 (d, J = 12.9 Hz), 128.40 (d, J = 31.1 Hz), 128.29 (d, J = 31.0 Hz), 124.21, 122.01, 48.90, 31.74 (d, J = 72.1 Hz), 21.54.

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):** δ 30.45.

**HRMS (ESI, m/z):** Calculated for C<sub>27</sub>H<sub>24</sub>N<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup>426.4678, found 426.2461

**6. 3-(diphenylphosphoryl)-1-(4-isopropylphenyl)-2-(pyridin-2-yl)propan-1-one (4af)**

A white solid, 54.4 mg, 60 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):** δ 8.26 (d, J = 2.8 Hz, 1H), 7.80 (d, J = 7.6 Hz, 2H), 7.64 (t, J = 8.8 Hz, 2H), 7.56 - 7.45 (m, 2H), 7.34 - 7.24 (m, 5H), 7.19 (d, J = 7.6 Hz, 3H), 7.10 (d, J = 8.0 Hz, 2H), 6.82 - 6.79 (m, 1H), 5.42 - 5.37 (m, 1H), 3.33 - 3.25 (m, 1H), 3.09 - 3.02 (m, 1H), 2.82 - 2.75 (m, 1H), 1.11 (d, J = 6.8 Hz, 6H).

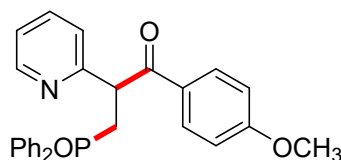
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):** δ 196.30 (d, J = 8.8 Hz), 157.76 (d, J = 5.9 Hz), 154.35, 149.57, 136.46, 133.43 (d, J = 97.7 Hz), 133.52, 132.44 (d, J = 97.1 Hz), 131.53 (d, J = 2.5 Hz), 131.09 (d, J = 2.7 Hz), 130.59 (d, J = 9.4 Hz), 130.53 (d, J = 9.5 Hz), 129.14, 128.31 (d, J = 27.3 Hz), 128.20 (d, J = 27.3 Hz), 126.39, 123.98, 121.90, 48.91, 34.01, 31.83 (d, J = 72.0 Hz), 23.41.

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):** δ 30.38.

**HRMS (ESI, m/z):** Calculated for C<sub>29</sub>H<sub>28</sub>N<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup>454.5218, found 454.2998

**7. 3-(diphenylphosphoryl)-1-(4-methoxyphenyl)-2-(pyridin-2-yl)propan-1-one (4ag)**

A white solid, 146.7 mg, 67 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):** δ 8.34 (d, J = 4.0 Hz, 1H), 7.92 (d, J = 8.0 Hz, 2H), 7.71 (t, J = 8.0 Hz, 2H), 7.57 (t, J = 8.8 Hz, 2H), 7.45 - 7.32 (m, 5H), 7.28 - 7.24 (m, 4H), 6.89 (t, J = 5.6 Hz, 1H), 6.80 (d, J = 8.0 Hz, 2H), 5.46 - 5.40 (m, 1H), 3.79 (s, 3H), 3.39 - 3.31 (m, 1H), 3.15 - 3.07 (m, 1H).

**<sup>13</sup>C NMR (101 MHz, Chloroform-d):** δ 195.29 (d, J = 9.2 Hz), 163.35, 157.95 (d, J = 5.9 Hz), 149.65, 136.55, 133.47 (d, J = 99.8 Hz), 132.46 (d, J = 99.1 Hz), 131.64 (d, J = 2.8 Hz), 131.33, 131.16 (d, J = 2.8 Hz), 130.68 (d, J = 9.4 Hz), 130.62 (d, J = 9.5

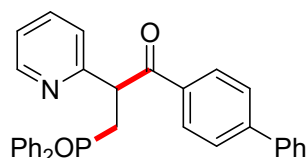
Hz), 128.69, 128.40 (d, J = 28.7 Hz), 128.28 (d, J = 28.7 Hz), 124.00, 121.98, 113.57, 55.37, 48.76, 31.86 (d, J = 72.0 Hz).

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):** δ 30.41.

**HRMS (ESI, m/z):** Calculated for C<sub>27</sub>H<sub>24</sub>N<sub>1</sub>O<sub>3</sub>P<sub>1</sub> (M+H)<sup>+</sup> 442.4668, found 442.1526

**8. 1-([1,1'-biphenyl]-4-yl)-3-(diphenylphosphoryl)-2-(pyridin-2-yl)propan-1-one (4ah)**

A white solid, 190.6 mg, 78 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):** δ 8.29 (d, J = 4.4 Hz, 1H), 7.98 (d, J = 7.6 Hz, 2H), 7.70 (dd, J = 10.4, 7.2 Hz, 2H), 7.56 (dd, J = 10.4, 7.6 Hz, 2H), 7.47 (t, J = 8.4 Hz, 4H), 7.35 - 7.26 (m, 9H), 7.20 (t, J = 7.2 Hz, 2H), 6.82 (t, J = 6.0 Hz, 1H), 5.52 - 5.46 (m, 1H), 3.44 - 3.35 (m, 1H), 3.14 - 3.07 (m, 1H).

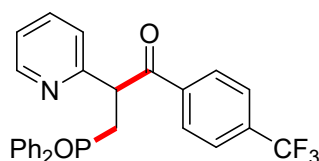
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):** δ 196.06 (d, J = 8.6 Hz), 157.49 (d, J = 6.2 Hz), 149.39, 145.16, 139.30, 136.31, 134.25, 133.23 (d, J = 99.7 Hz), 132.25 (d, J = 98.4 Hz), 131.35 (d, J = 2.5 Hz), 130.93 (d, J = 2.7 Hz), 130.36 (d, J = 9.5 Hz), 130.30 (d, J = 9.6 Hz), 129.24, 128.53, 128.13 (d, J = 25.7 Hz), 128.02 (d, J = 25.7 Hz), 127.84, 126.73 (d, J = 12.9 Hz), 123.73, 121.76, 48.95, 31.64 (d, J = 71.9 Hz).

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):** δ 30.16.

**HRMS (ESI, m/z):** Calculated for C<sub>32</sub>H<sub>26</sub>N<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup> 488.5388, found 488.3201

**9. 3-(diphenylphosphoryl)-2-(pyridin-2-yl)-1-(4-(trifluoromethyl)phenyl)propan-1-one (4ai)**

A yellow solid, 105.2 mg, 44 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):** δ 8.30 (d, J = 4.4 Hz, 1H), 7.97 (d, J = 8.0 Hz, 2H), 7.78 (dd, J = 10.4, 7.6 Hz, 2H), 7.55 (t, J = 5.2 Hz, 4H), 7.40 - 7.30 (m, 5H), 7.24 (t, J = 6.0 Hz, 3H), 6.88 (t, J = 6.0 Hz, 1H), 5.44 - 5.38 (m, 1H), 3.43 - 3.35 (m, 1H), 3.06 - 2.99 (m, 1H).

**<sup>13</sup>C NMR (101 MHz, Chloroform-d):** δ 196.05 (d, J = 8.4 Hz), 157.20 (d, J = 6.5 Hz), 149.74, 138.65, 136.76, 133.96 (q, J = 32.9 Hz), 133.74, 132.74, 132.98 (d, J = 101.6 Hz), 131.94 (d, J = 101.7 Hz), 131.71 (d, J = 2.7 Hz), 131.30 (d, J = 2.7 Hz), 130.59 (d, J = 9.4 Hz), 130.54 (d, J = 9.6 Hz), 129.13, 128.44 (d, J = 24.1 Hz), 128.33 (d, J = 24.2 Hz), 125.33 (q, J = 3.7 Hz), 124.04, 123.41 (q, J = 273.7 Hz), 122.23, 49.61, 31.78 (d, J = 71.7 Hz).

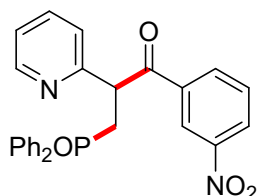
**<sup>31</sup>P NMR (162 MHz, Chloroform-d):** δ 30.16.

**<sup>19</sup>F NMR (376 MHz, Chloroform-d):**  $\delta$  -63.20.

**HRMS (ESI, m/z):** Calculated for C<sub>27</sub>H<sub>21</sub>F<sub>3</sub>N<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup>480.4390, found 480.2675

**10. 3-(diphenylphosphoryl)-1-(3-nitrophenyl)-2-(pyridin-2-yl)propan-1-one (4aj)**

A yellow solid, 182.3 mg, 80 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):**  $\delta$  8.63 (s, 1H), 8.30 (s, 1H), 8.22 (t, J = 8.2 Hz, 2H), 7.66 (t, J = 9.2 Hz, 2H), 7.56 - 7.47 (m, 3H), 7.41 - 7.34 (m, 5H), 7.28 - 7.22 (m, 4H), 6.90 (s, 1H), 5.43 - 5.37 (m, 1H), 3.46 - 3.37 (m, 1H), 3.02 - 2.95 (m, 1H).

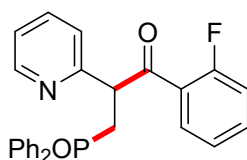
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):**  $\delta$  194.99 (d, J = 8.1 Hz), 157.05 (d, J = 6.8 Hz), 149.92, 148.16, 137.20, 137.02, 134.45, 133.11 (d, J = 100.7 Hz), 132.09 (d, J = 99.6 Hz), 131.86 (d, J = 2.7 Hz), 131.48 (d, J = 2.7 Hz), 130.64 (d, J = 9.5 Hz), 130.61 (d, J = 9.7 Hz), 129.63, 128.57 (d, J = 20.5 Hz), 128.45 (d, J = 20.5 Hz), 127.09, 124.09, 123.71, 122.46, 49.73, 31.88 (d, J = 71.6 Hz).

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):**  $\delta$  30.02.

**HRMS (ESI, m/z):** Calculated for C<sub>26</sub>H<sub>21</sub>N<sub>2</sub>O<sub>4</sub>P<sub>1</sub> (M+H)<sup>+</sup>457.4378, found 457.2421

**11. 3-(diphenylphosphoryl)-1-(2-fluorophenyl)-2-(pyridin-2-yl)propan-1-one (4ak)**

A yellow solid, 186.8 mg, 87 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):**  $\delta$  8.27 (d, J = 4.0 Hz, 1H), 7.77 - 7.68 (m, 3H), 7.54 (t, J = 8.8 Hz, 2H), 7.44 - 7.40 (m, 3H), 7.32 (t, J = 7.6 Hz, 2H), 7.26 - 7.21 (m, 4H), 7.06 (t, J = 7.6 Hz, 1H), 6.91 (t, J = 10 Hz, 1H), 6.83 (d, J = 1.6 Hz, 1H), 5.35 - 5.30 (m, 1H), 3.49 - 3.41 (m, 1H), 3.11 - 3.03 (m, 1H).

**<sup>13</sup>C NMR (101 MHz, Chloroform-d):**  $\delta$  195.38 (d, J = 10.4 Hz), 160.22 (d, J = 255.4 Hz), 156.82 (d, J = 4.8 Hz), 149.30, 136.20, 133.92 (d, J = 9.0 Hz), 133.56 (d, J = 100.2 Hz), 132.30 (d, J = 99.2 Hz), 131.47 (d, J = 2.6 Hz), 130.95, 130.42 (d, J = 9.1 Hz), 130.33 (d, J = 9.2 Hz), 128.69 (d, J = 12.9 Hz), 128.24 (d, J = 37.3 Hz), 128.12 (d, J = 37.3 Hz), 125.15 (d, J = 12.9 Hz), 125.01, 124.05 (d, J = 3.3 Hz), 121.91, 116.07 (d, J = 23.6 Hz), 52.68 (d, J = 6.0 Hz), 31.13 (d, J = 72.7 Hz).

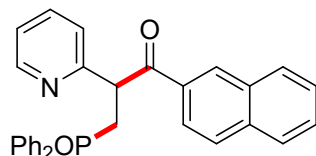
**<sup>31</sup>P NMR (162 MHz, Chloroform-d):**  $\delta$  30.15.

**<sup>19</sup>F NMR (376 MHz, Chloroform-d):**  $\delta$  -109.46.

**HRMS (ESI, m/z):** Calculated for C<sub>26</sub>H<sub>21</sub>F<sub>1</sub>N<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup> 430.4312, found 430.2232

**12. 3-(diphenylphosphoryl)-1-(naphthalen-2-yl)-2-(pyridin-2-yl)propan-1-one (4al)**

A white solid, 207 mg, 90 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):**  $\delta$  8.43 (s, 1H), 8.27 (d, J = 4.8 Hz, 1H), 7.87 (d, J = 8.6 Hz, 1H), 7.81 (d, J = 8.0 Hz, 1H), 7.72 – 7.64 (m, 4H), 7.53 (dd, J = 11.7, 7.6 Hz, 2H), 7.49 – 7.40 (m, 2H), 7.35 - 7.18 (m, 9H), 6.80 (q, J = 4.4 Hz, 1H), 5.61 – 5.55 (m, 1H), 3.41 – 3.33 (m, 1H), 3.14 – 3.06 (m, 1H).

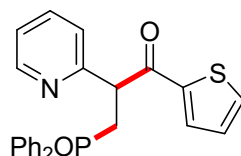
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):**  $\delta$  196.76 (d, J = 8.9 Hz), 157.60 (d, J = 5.7 Hz), 149.65, 136.57, 135.30, 133.26 (d, J = 100.2 Hz), 133.03, 132.21 (d, J = 99.4 Hz), 132.20, 131.64 (d, J = 2.7 Hz), 131.18 (d, J = 2.8 Hz), 130.83, 130.61 (d, J = 9.5 Hz), 130.54 (d, J = 9.6 Hz), 129.60, 128.40 (t, J = 12.2 Hz), 128.38 (d, J = 28.5 Hz), 128.14 (d, J = 2.1 Hz), 127.48, 126.51, 124.41, 124.10, 122.00, 49.07, 31.79 (d, J = 72.0 Hz).

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):**  $\delta$  30.54.

**HRMS (ESI, m/z):** Calculated for C<sub>26</sub>H<sub>21</sub>Cl<sub>1</sub>N<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup> 462.1545, found 462.1618.

**13. 3-(diphenylphosphoryl)-2-(pyridin-2-yl)-1-(thiophen-2-yl)propan-1-one (4am)**

A white solid, 73 mg, 35 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):**  $\delta$  8.26 (d, J = 4.1 Hz, 1H), 7.67 - 7.55 (m, 3H), 7.50 - 7.44 (m, 2H), 7.41 (dd, J = 5.0, 1.1 Hz, 1H), 7.34 - 7.21 (m, 5H), 7.20 - 7.12 (m, 3H), 6.90 - 6.85 (m, 1H), 6.85 - 6.79 (m, 1H), 5.22 - 5.16 (m, 1H), 3.29 - 3.21 (m, 1H), 3.05 - 2.98 (m, 1H).

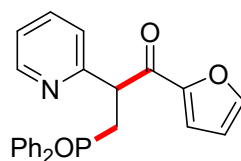
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):**  $\delta$  189.47 (d, J = 9.0 Hz), 157.35, 149.61, 142.74, 136.53, 134.13, 133.30, 133.08 (d, J = 77.8 Hz), 132.09 (d, J = 77.2 Hz), 131.63 (d, J = 2.7 Hz), 131.20 (d, J = 2.8 Hz), 130.57 (d, J = 9.4 Hz), 130.56 (d, J = 9.6 Hz), 128.34 (d, J = 24.1 Hz), 128.22 (d, J = 24.1 Hz), 127.93, 123.83, 122.16, 50.15, 31.61 (d, J = 71.8 Hz).

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):**  $\delta$  30.23.

**HRMS (ESI, m/z):** Calculated for C<sub>24</sub>H<sub>20</sub>N<sub>1</sub>O<sub>2</sub>P<sub>1</sub>S<sub>1</sub> (M+H)<sup>+</sup> 418.0953, found 418.1042.

**14. 3-(diphenylphosphoryl)-1-(furan-2-yl)-2-(pyridin-2-yl)propan-1-one (4an)**

A brown solid, 56 mg, 56 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):**  $\delta$  8.33 (d,  $J$  = 4.4 Hz, 1H), 7.69 (t,  $J$  = 8.0 Hz, 2H), 7.55 (t,  $J$  = 8.0 Hz, 2H), 7.42 - 7.30 (m, 6H), 7.26 - 7.22 (m, 3H), 7.13 (s, 1H), 6.90 (t,  $J$  = 6.0 Hz, 1H), 6.36 (d,  $J$  = 1.2 Hz, 1H), 5.24 - 5.18 (m, 1H), 3.36 - 3.28 (m, 1H), 3.14 - 3.06 (m, 1H).

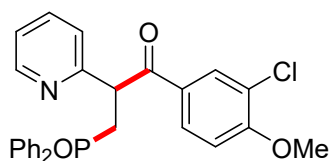
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):**  $\delta$  185.18 (d,  $J$  = 9.0 Hz), 157.04 (d,  $J$  = 5.9 Hz), 151.41, 149.48, 146.45, 136.33, 133.14 (d,  $J$  = 100.0 Hz), 132.33 (d,  $J$  = 99.5 Hz), 131.54 (d,  $J$  = 2.6 Hz), 131.12 (d,  $J$  = 2.7 Hz), 130.56 (d,  $J$  = 9.5 Hz), 130.50 (d,  $J$  = 9.5 Hz), 128.26 (d,  $J$  = 24.2 Hz), 128.15 (d,  $J$  = 24.3 Hz), 124.21, 122.02, 118.59, 112.13, 49.09, 30.88 (d,  $J$  = 71.8 Hz).

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):**  $\delta$  30.14.

**HRMS (ESI, m/z):** Calculated for C<sub>24</sub>H<sub>20</sub>N<sub>1</sub>O<sub>3</sub>P<sub>1</sub> (M+H)<sup>+</sup>402.4018, found 402.1883

#### 15. 1-(3-chloro-4-methoxyphenyl)-3-(diphenylphosphoryl)-2-(pyridin-2-yl)propan-1-one (4ao)

A white solid, 78 mg, 82 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):**  $\delta$  8.34 (d,  $J$  = 4.2 Hz, 1H), 7.92 (d,  $J$  = 2.1 Hz, 1H), 7.89 (dd,  $J$  = 8.6, 2.1 Hz, 1H), 7.70 (dd,  $J$  = 11.6, 6.9 Hz, 2H), 7.57 (dd,  $J$  = 11.6, 7.1 Hz, 2H), 7.43 (d,  $J$  = 5.9 Hz, 1H), 7.40 - 7.34 (m, 4H), 7.30 - 7.24 (m, 3H), 6.93 - 6.90 (m, 1H), 6.83 (d,  $J$  = 8.6 Hz, 1H), 5.43 - 5.37 (m, 1H), 3.89 (s, 3H), 3.40 - 3.32 (m, 1H), 3.07 - 3.04 (m, 1H).

**<sup>13</sup>C NMR (101 MHz, Chloroform-d):**  $\delta$  194.48 (d,  $J$  = 8.5 Hz), 158.53, 157.60 (d,  $J$  = 6.3 Hz), 149.71, 136.64, 133.25 (d,  $J$  = 96.8 Hz), 132.26 (d,  $J$  = 96.1 Hz), 131.66 (d,  $J$  = 2.7 Hz), 131.23 (d,  $J$  = 2.9 Hz), 131.06, 130.60 (d,  $J$  = 9.5 Hz), 130.58 (d,  $J$  = 9.6 Hz), 129.53, 129.22, 128.40 (d,  $J$  = 24.5 Hz), 128.29 (d,  $J$  = 24.2 Hz), 123.83, 122.71, 122.10, 110.93, 56.23, 48.83, 31.85 (d,  $J$  = 71.9 Hz).

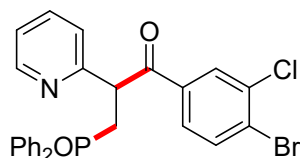
**<sup>31</sup>P NMR (162 MHz, Chloroform-d):**  $\delta$  30.29.

**HRMS (ESI, m/z):** Calculated for C<sub>27</sub>H<sub>28</sub>ClN<sub>1</sub>O<sub>3</sub>P<sub>1</sub> (M+H)<sup>+</sup>476.1104, found 476.1178.

#### 16. 1-(4-bromo-3-chlorophenyl)-3-(diphenylphosphoryl)-2-(pyridin-2-yl)propan-1-one (4ap)

A white solid, 64 mg, 61 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)





**<sup>1</sup>H NMR (400 MHz, Chloroform-d):** δ 8.22 (d, J = 4.0 Hz, 1H), 7.82 (d, J = 2.0 Hz, 1H), 7.60 - 7.54 (m, 2H), 7.52 (d, J = 2.1 Hz, 1H), 7.48 - 7.42 (m, 3H), 7.34 - 7.29 (m, 1H), 7.28 - 7.22 (m, 4H), 7.18 - 7.12 (m, 3H), 6.80 (ddd, J = 7.6, 4.8, 1.2 Hz, 1H), 5.27 - 5.21 (m, 1H), 3.32 - 3.23 (m, 1H), 2.95 - 2.87 (m, 1H).

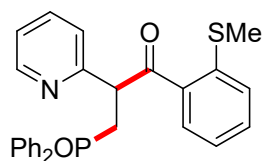
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):** δ 194.89 (d, J = 8.4 Hz), 156.98 (d, J = 6.5 Hz), 149.71, 136.74, 135.93, 134.92, 133.65, 133.03 (d, J = 100.4 Hz), 132.00 (d, J = 99.5 Hz), 131.67 (d, J = 2.8 Hz), 131.26 (d, J = 2.8 Hz), 130.48 (d, J = 9.5 Hz), 130.45 (d, J = 9.6 Hz), 130.36, 128.38 (d, J = 23.2 Hz), 128.26 (d, J = 23.2 Hz), 127.87, 127.77, 123.83, 122.23, 49.26 (d, J = 1.3 Hz), 31.70 (d, J = 71.6 Hz).

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):** δ 30.07.

**HRMS (ESI, m/z):** Calculated for C<sub>26</sub>H<sub>26</sub>BrClN<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup>524.0104, found 524.0177.

#### 17. 3-(diphenylphosphoryl)-1-(2-(methylthio)phenyl)-2-(pyridin-2-yl)propan-1-one (4aq)

A white solid, 96 mg, 42 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):** δ 8.25 (d, J = 4.0 Hz, 1H), 7.78 (d, J = 7.9 Hz, 1H), 7.70 - 7.65 (m, 2H), 7.52 - 7.46 (m, 2H), 7.37 - 7.32 (m, 3H), 7.27 - 7.17 (m, 5H), 7.12 (t, J = 7.4 Hz, 2H), 6.97 (t, J = 8.0 Hz, 1H), 6.80 (ddd, J = 7.5, 4.9, 1.2 Hz, 1H), 5.42 - 5.36 (m, 1H), 3.37 - 3.29 (m, 1H), 3.15 - 3.07 (m, 1H), 2.27 (s, 3H).

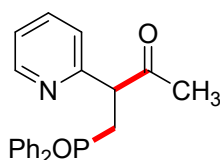
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):** δ 197.53 (d, J = 10.0 Hz), 157.13 (d, J = 4.8 Hz), 149.43, 142.62, 136.36, 133.28 (d, J = 100.4 Hz), 133.51, 131.82, 132.15 (d, J = 99.4 Hz), 131.50 (d, J = 2.7 Hz), 131.01 (d, J = 2.8 Hz), 130.45 (d, J = 19.4 Hz), 130.45, 128.29 (d, J = 38.2 Hz), 128.17 (d, J = 38.4 Hz), 125.17, 123.96, 123.29, 121.91, 50.28, 31.41 (d, J = 72.4 Hz), 15.99.

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):** δ 30.53.

**HRMS (ESI, m/z):** Calculated for C<sub>27</sub>H<sub>27</sub>Cl<sub>1</sub>N<sub>1</sub>O<sub>2</sub>P<sub>1</sub>S<sub>1</sub> (M+H)<sup>+</sup>458.1268, found 458.1338.

#### 18. 4-(diphenylphosphoryl)-3-(pyridin-2-yl)butan-2-one (4ar)

A yellow oil, 110.2 mg, 63 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):**  $\delta$  8.40 (d,  $J$  = 4.5 Hz, 1H), 7.81 - 7.62 (m, 2H), 7.60 - 7.33 (m, 6H), 7.31 (s, 1H), 7.28 - 7.14 (m, 3H), 7.06 - 6.91 (m, 1H), 4.57 - 4.27 (m, 1H), 3.40 - 3.25 (m, 1H), 2.93 (dt,  $J$  = 15.7, 8.0 Hz, 1H), 1.99 (s, 3H).

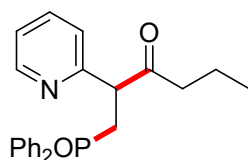
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):**  $\delta$  204.12 (d,  $J$  = 9.8 Hz), 157.14 (d,  $J$  = 4.7 Hz), 149.59, 136.49, 133.44 (d,  $J$  = 100.2 Hz), 132.18 (d,  $J$  = 99.0 Hz), 131.51 (d,  $J$  = 2.8 Hz), 130.96 (d,  $J$  = 2.9 Hz), 130.41 (d,  $J$  = 12.3 Hz), 130.32 (d,  $J$  = 12.4 Hz), 128.23 (d,  $J$  = 40.9 Hz), 128.11 (d,  $J$  = 41.1 Hz), 124.49, 122.08, 54.43, 30.06 (d,  $J$  = 72.6 Hz), 28.23.

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):**  $\delta$  30.40.

**HRMS (ESI,  $m/z$ ):** Calculated for  $C_{21}H_{20}N_1O_2P_1$  ( $M+H$ )<sup>+</sup> 350.3698, found 350.1446

### 19. 1-(diphenylphosphoryl)-2-(pyridin-2-yl)hexan-3-one (4as)

A yellow oil, 144.1 mg, 76 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 %  $Et_3N$ )



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):**  $\delta$  8.41 (d,  $J$  = 4.4 Hz, 1H), 7.75 (dd,  $J$  = 10.8, 7.6 Hz, 2H), 7.56 - 7.40 (m, 6H), 7.35 (t,  $J$  = 7.2 Hz, 1H), 7.26 (t,  $J$  = 7.2 Hz, 2H), 7.21 (d,  $J$  = 7.6 Hz, 1H), 7.00 (t,  $J$  = 6.0 Hz, 1H), 4.50 - 4.42 (m, 2H), 3.39 - 3.31 (m, 1H), 2.97 - 2.89 (m, 1H), 2.28 - 2.23 (m, 2H), 1.48 - 1.40 (m, 2H), 0.72 (t,  $J$  = 7.6 Hz, 3H).

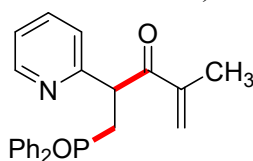
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):**  $\delta$  206.49 (d,  $J$  = 9.1 Hz), 157.46 (d,  $J$  = 5.2 Hz), 149.67, 136.56, 133.55 (d,  $J$  = 100.0 Hz), 132.45 (d,  $J$  = 99.1 Hz), 131.61 (d,  $J$  = 2.5 Hz), 131.09 (d,  $J$  = 2.6 Hz), 130.58 (d,  $J$  = 9.4 Hz), 130.52 (d,  $J$  = 9.5 Hz), 128.36 (d,  $J$  = 39.5 Hz), 128.24 (d,  $J$  = 39.5 Hz), 124.60, 122.13, 53.72, 43.10, 30.24 (d,  $J$  = 72.7 Hz), 17.00, 13.38.

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):**  $\delta$  30.39.

**HRMS (ESI,  $m/z$ ):** Calculated for  $C_{23}H_{24}N_1O_2P_1$  ( $M+H$ )<sup>+</sup> 378.4238, found 378.2216

### 20. 5-(diphenylphosphoryl)-2-methyl-4-(pyridin-2-yl)pent-1-en-3-one (4at)

A black oil, 121.5 mg, 65 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 %  $Et_3N$ )



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):**  $\delta$  8.38 (d,  $J$  = 3.6 Hz, 1H), 7.73 (t,  $J$  = 8.4 Hz, 2H), 7.58 (t,  $J$  = 8.8 Hz, 2H), 7.48 - 7.34 (m, 5H), 7.28 (d,  $J$  = 7.2 Hz, 2H), 7.19 (d,  $J$

= 8.0 Hz, 1H), 6.95 (t, J = 6.0 Hz, 1H), 6.02 (s, 1H), 5.67 (s, 1H), 5.24 - 5.18 (m, 1H), 3.33 - 3.25 (m, 1H), 3.03 - 2.95 (m, 1H), 1.74 (s, 3H).

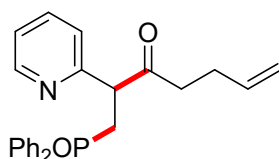
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):** δ 198.15 (d, J = 8.3 Hz), 157.94 (d, J = 6.5 Hz), 149.40, 142.98, 136.41, 133.26 (d, J = 99.9 Hz), 132.24 (d, J = 99.2 Hz), 131.48 (d, J = 2.7 Hz), 131.05 (d, J = 2.7 Hz), 130.48, 130.39, 128.26 (d, J = 28.4 Hz), 128.14 (d, J = 28.4 Hz), 125.93, 123.65, 121.80, 47.99, 31.64 (d, J = 72.0 Hz), 17.96.

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):** δ 30.14.

**HRMS (ESI, m/z):** Calculated for C<sub>23</sub>H<sub>22</sub>N<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup>376.4078, found 376.1852

### 21. 1-(diphenylphosphoryl)-2-(pyridin-2-yl)hept-6-en-3-one (4au)

A light yellow solid, 131.2 mg, 67 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):** δ 8.32 (d, J = 4.0 Hz, 1H), 7.66 (t, J = 8.0 Hz, 2H), 7.47 - 7.31 (m, 6H), 7.26 (t, J = 7.2 Hz, 1H), 7.17 (t, J = 7.2 Hz, 1H), 7.12 (d, J = 7.6 Hz, 3H), 6.92 (t, J = 5.6 Hz, 1H), 5.58 - 5.48 (m, 1H), 4.79 (d, J = 9.6 Hz, 2H), 4.75 (s, 1H), 4.42 - 4.36 (m, 1H), 3.31 - 3.23 (m, 1H), 2.88 - 2.80 (m, 1H), 2.29 (t, J = 7.6 Hz, 2H), 2.12 - 2.06 (m, 2H).

**<sup>13</sup>C NMR (101 MHz, Chloroform-d):** δ 205.68 (d, J = 9.2 Hz), 157.17 (d, J = 5.1 Hz), 149.56, 136.67, 136.59, 133.35 (d, J = 100.1 Hz), 132.18 (d, J = 99.2 Hz), 131.62 (d, J = 2.7 Hz), 131.08 (d, J = 2.8 Hz), 130.50 (d, J = 9.5 Hz), 130.44 (d, J = 9.5 Hz), 128.33 (d, J = 39.6 Hz), 128.21 (d, J = 39.7 Hz), 124.58, 122.18, 114.95, 53.68 (d, J = 1.0 Hz), 40.25, 30.16 (d, J = 72.7 Hz), 27.46.

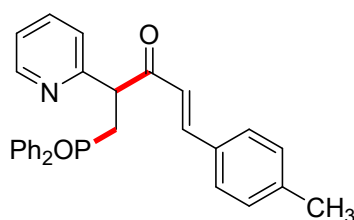
**<sup>31</sup>P NMR (162 MHz, Chloroform-d):** δ 30.40.

**HRMS (ESI, m/z):** Calculated for C<sub>24</sub>H<sub>24</sub>N<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup>390.4348, found 390.2124

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):** δ 30.71.

### 22. 5-(diphenylphosphoryl)-4-(pyridin-2-yl)-1-(p-tolyl)pent-1-en-3-one (4av)

A yellow solid, 74 mg, 82 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):** δ 8.34 (d, J = 4.6 Hz, 1H), 7.71 (dd, J = 11.5, 7.1 Hz, 2H), 7.56 - 7.49 (m, 3H), 7.40 - 7.31 (m, 4H), 7.28 - 7.22 (m, 4H), 7.20 - 7.12 (m, 2H), 7.06 (d, J = 7.8 Hz, 2H), 6.91 - 6.88 (m, 1H), 6.55 (d, J = 15.9 Hz, 1H), 4.86 - 4.80 (m, 1H), 3.34 - 3.26 (m, 1H), 3.07 - 2.99 (m, 1H), 2.27 (s, 3H).

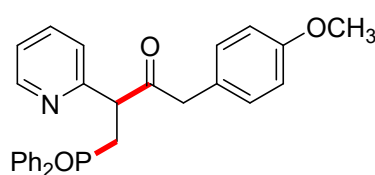
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):**  $\delta$  195.65 (d, J = 9.8 Hz), 157.28 (d, J = 4.9 Hz), 149.81, 143.79, 141.07, 136.66, 133.32 (d, J = 99.9 Hz), 131.73 (d, J = 2.6 Hz), 131.37 (d, J = 96.0 Hz), 131.51, 131.18 (d, J = 2.6 Hz), 130.87 (d, J = 9.6 Hz), 130.64 (d, J = 9.5 Hz), 129.50, 128.41, 128.43 (d, J = 37.0 Hz), 128.31 (d, J = 38.0 Hz), 124.51, 123.03, 122.21, 52.35, 30.66 (d, J = 74.7 Hz), 21.46.

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):**  $\delta$  30.17.

**HRMS (ESI, m/z):** Calculated for C<sub>29</sub>H<sub>26</sub>N<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup>452.1775, found 452.1701

**23. 4-(diphenylphosphoryl)-1-(4-methoxyphenyl)-3-(pyridin-2-yl)butan-2-one (4aw)**

A white solid, 56.5 mg, 62 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):**  $\delta$  8.41 (d, J = 4.0 Hz, 1H), 7.71 (t, J = 8.0 Hz, 2H), 7.54 - 7.50 (m, 3H), 7.45 - 7.33 (m, 4H), 7.26 (t, J = 7.2 Hz, 2H), 7.14 (d, J = 7.6 Hz, 1H), 7.01 (t, J = 5.6 Hz, 1H), 6.84 (d, J = 7.6 Hz, 2H), 6.75 (d, J = 7.6 Hz, 2H), 4.60 - 4.54 (m, 1H), 3.77 (s, 3H), 3.52 (dd, J = 28.4, 16 Hz, 2H), 3.36 - 3.29 (m, 1H), 2.94 - 2.86 (m, 1H).

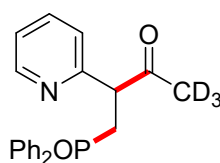
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):**  $\delta$  204.48 (d, J = 9.2 Hz), 158.45, 157.26 (d, J = 5.0 Hz), 149.71, 136.61, 133.40 (d, J = 100.3 Hz), 132.38 (d, J = 99.5 Hz), 131.66 (d, J = 2.6 Hz), 131.15 (d, J = 2.7 Hz), 130.58 (d, J = 9.5 Hz), 130.54 (d, J = 9.5 Hz), 130.43, 128.41 (d, J = 38.5 Hz), 128.30 (d, J = 38.3 Hz), 125.67, 124.91, 122.24, 113.88, 55.16, 52.92, 47.19, 30.46 (d, J = 72.7 Hz).

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):**  $\delta$  30.71.

**HRMS (ESI, m/z):** Calculated for C<sub>28</sub>H<sub>26</sub>N<sub>1</sub>O<sub>3</sub>P<sub>1</sub> (M+H)<sup>+</sup>456.4938, found 456.2837

**24. 4-(diphenylphosphoryl)-3-(pyridin-2-yl)butan-2-one-1,1,1-d<sub>3</sub> (4ax)**

A brown oil, 118.8 mg, 68 %, H/D = 10/90, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):**  $\delta$  8.42 (d, J = 2.8 Hz, 1H), 7.75 (t, J = 8.0 Hz, 2H), 7.55 - 7.40 (m, 6H), 7.35 (t, J = 7.2 Hz, 1H), 7.26 (t, J = 7.2 Hz, 2H), 7.20 (d, J = 8.0 Hz, 1H), 7.02 (t, J = 5.6 Hz, 1H), 4.50 - 4.45 (m, 1H), 3.38 - 3.30 (m, 1H), 2.98 - 2.90 (m, 1H), 1.98 (s, 0.4H, H/D = 11:89).

**<sup>13</sup>C NMR (101 MHz, Chloroform-d):**  $\delta$  204.42 (d, J = 9.5 Hz), 157.11 (d, J = 4.5 Hz), 149.68, 136.60, 133.41 (d, J = 100.4 Hz), 132.09 (d, J = 98.6 Hz), 131.62 (d, J = 2.9 Hz), 131.05 (d, J = 2.7 Hz), 130.47 (d, J = 12.7 Hz), 130.38 (d, J = 12.7 Hz),

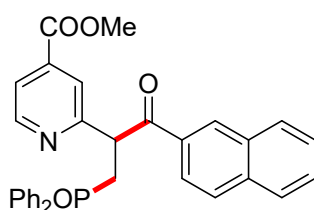
128.31 (d, J = 43.1 Hz), 128.19 (d, J = 43.2 Hz), 124.68, 122.18, 54.25, 30.03 (d, J = 73.0 Hz), 28.05-27.41 (m).

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):** δ 30.50.

**HRMS (ESI, m/z):** Calculated for C<sub>21</sub>H<sub>17</sub>D<sub>3</sub>N<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup> 353.3881, found 353.1669.

### 25. Methyl 2-(3-(diphenylphosphoryl)-1-(naphthalen-2-yl)-1-oxopropan-2-yl)isonicotinate (4ba)

A white solid, 39 mg, 38%, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):** δ 8.45 (s, 1H), 8.40 (d, J = 5.0 Hz, 1H), 7.88 (dd, J = 8.6 Hz, 1.8 Hz, 1H), 7.83 (d, J = 8.0 Hz, 1H), 7.75 (s, 1H), 7.72 - 7.64 (m, 4H), 7.52 - 7.41 (m, 4H), 7.37 - 7.29 (m, 4H), 7.23 (dd, J = 7.5 Hz, 1.8 Hz, 1H), 7.20 - 7.14 (m, 2H), 5.71 - 5.65 (m, 1H), 3.82 (s, 3H), 3.39 - 3.31 (m, 1H), 3.14 - 3.06 (m, 1H).

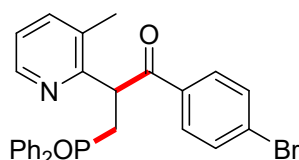
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):** δ 196.48 (d, J = 9.6 Hz), 164.93, 158.78 (d, J = 5.1 Hz), 150.44, 137.80, 135.43, 133.22 (d, J = 100.5 Hz), 132.81, 132.26, 132.16 (d, J = 99.2 Hz), 131.80 (d, J = 2.7 Hz), 131.21 (d, J = 2.8 Hz), 131.02, 130.65 (d, J = 9.6 Hz), 130.51 (d, J = 9.7 Hz), 129.72, 128.59, 128.49 (d, J = 29.2 Hz), 128.33 (d, J = 36.9 Hz), 128.27, 127.55, 126.66, 124.39, 123.37, 121.34, 52.60, 49.09, 31.99 (d, J = 72.0 Hz).

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):** δ 29.92.

**HRMS (ESI, m/z):** Calculated for C<sub>28</sub>H<sub>23</sub>BrN<sub>1</sub>O<sub>4</sub>P<sub>1</sub> (M+H)<sup>+</sup> 520.1599, found 520.1674.

### 26. 1-(4-bromophenyl)-3-(diphenylphosphoryl)-2-(3-methylpyridin-2-yl)propan-1-one (4bb)

A white solid, 206 mg, 82%, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):** δ 8.00 (d, J = 4.7 Hz, 1H), 7.55 - 7.50 (m, 2H), 7.45 (d, J = 8.2 Hz, 2H), 7.35 - 7.31 (m, 2H), 7.26 - 7.20 (m, 5H), 7.13 - 7.03 (m, 3H), 6.91 (d, J = 7.3 Hz, 1H), 6.61 (dd, J = 7.3, 4.8 Hz, 1H), 5.25 - 5.19 (m, 1H), 3.19 - 3.11 (m, 1H), 2.96 - 2.90 (m, 1H), 2.23 (s, 3H).

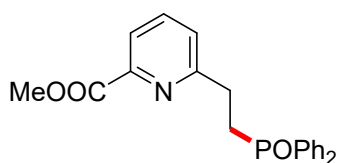
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):**  $\delta$  196.30 (d,  $J = 10.0$  Hz), 167.67, 155.79 (d,  $J = 4.5$  Hz), 147.23, 138.53, 135.13, 132.03 (d,  $J = 9.8$  Hz), 131.72 (d,  $J = 2.2$  Hz), 131.69, 131.40, 131.18 (d,  $J = 2.7$  Hz), 130.42 (dd,  $J = 9.6, 5.7$  Hz), 129.82 (d,  $J = 211.9$  Hz), 129.75, 128.55 (d,  $J = 11.9$  Hz), 128.08 (d,  $J = 11.9$  Hz), 127.73, 122.35, 45.76 (d,  $J = 54.2$  Hz), 31.81 - 29.65 (m), 19.04.

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):**  $\delta$  30.51.

**HRMS (ESI, m/z):** Calculated for C<sub>27</sub>H<sub>23</sub>Br<sub>1</sub>N<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup> 504.0650, found 504.0724.

### 27. Methyl 6-(2-(diphenylphosphoryl)ethyl)picolinate (4bc)

A white solid, 122 mg, 67%, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):**  $\delta$  7.73 (d,  $J = 7.2$  Hz, 1H), 7.63 - 7.58 (m, 4H), 7.50 (t,  $J = 7.7$  Hz, 1H), 7.35 - 7.25 (m, 6H), 7.17 (dd,  $J = 8.4$  Hz, 1H), 3.82 (s, 3H), 3.13 - 3.06 (m, 2H), 2.71 - 2.62 (m, 2H).

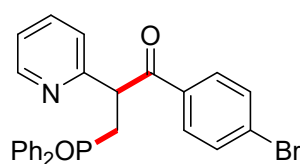
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):**  $\delta$  165.56, 160.59 (d,  $J = 13.3$  Hz), 147.36, 137.17, 132.96, 131.98, 131.60, 131.57, 130.68, 130.59, 128.51, 128.39, 126.37, 122.90, 52.66, 29.62 (d,  $J = 3.0$  Hz), 28.99 (d,  $J = 71.6$  Hz).

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):**  $\delta$  31.15.

**HRMS (ESI, m/z):** Calculated for C<sub>21</sub>H<sub>20</sub>Cl<sub>1</sub>N<sub>1</sub>O<sub>3</sub>P<sub>1</sub> (M+H)<sup>+</sup> 366.1181, found 366.1254.

### 28. 1-(4-bromophenyl)-3-(diphenylphosphoryl)-2-(pyridin-2-yl)propan-1-one (4bd)

A white solid, 100.1 mg, 40 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N).



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):**  $\delta$  8.34 (d,  $J = 4.2$  Hz, 1H), 7.80 - 7.75 (m, 2H), 7.70 (dd,  $J = 11.5, 7.0$  Hz, 2H), 7.57 (dd,  $J = 11.3, 7.9$  Hz, 2H), 7.48 - 7.32 (m, 7H), 7.26 (s, 3H), 6.92 (dd,  $J = 7.5, 4.9$  Hz, 1H), 5.41 - 5.35 (m, 1H), 3.42 - 3.34 (m, 1H), 3.12 - 3.02 (m, 1H).

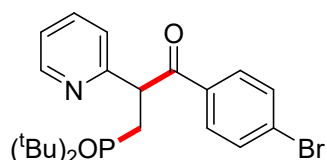
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):**  $\delta$  195.77 (d,  $J = 8.7$  Hz), 157.26 (d,  $J = 6.1$  Hz), 149.64, 136.60, 134.36, 133.19 (d,  $J = 100.3$  Hz), 132.12 (d,  $J = 99.5$  Hz), 131.61 (d,  $J = 2.8$  Hz), 131.54, 131.17 (d,  $J = 2.7$  Hz), 130.49 (d,  $J = 9.4$  Hz), 130.43 (d,  $J = 9.6$  Hz), 130.30, 128.34 (d,  $J = 26.6$  Hz), 128.22 (d,  $J = 26.5$  Hz), 128.03, 123.90, 122.07, 49.13, 32.64 (d,  $J = 71.9$  Hz).

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):** δ 30.12.

**HRMS (ESI, m/z):** Calculated for C<sub>26</sub>H<sub>21</sub>BrN<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup>491.3368, found 491.2162.

**29. 1-(4-bromophenyl)-3-(di-tert-butylphosphoryl)-2-(pyridin-2-yl)propan-1-one (4be)**

A white solid, 200 mg, 89 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):** δ 8.50 (d, J = 4.1 Hz, 1H), 7.88 (d, J = 8.6 Hz, 2H), 7.59 (td, J = 7.7, 1.7 Hz, 1H), 7.50 (d, J = 8.6 Hz, 2H), 7.44 (d, J = 7.8 Hz, 1H), 7.12 - 7.10 (m, 1H), 5.36 - 5.33 (m, 1H), 3.21 - 3.13 (m, 1H), 2.17 - 2.10 (m, 1H), 1.25 (d, J = 13.4 Hz, 9H). 1.19 (d, J = 13.2 Hz, 9H).

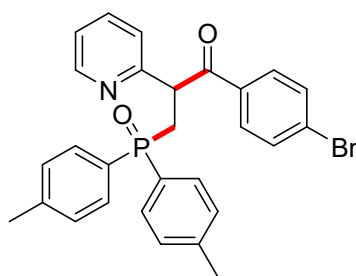
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):** δ 196.97 (d, J = 3.6 Hz), 159.09 (d, J = 8.0 Hz), 149.91, 137.00, 135.53, 131.69, 130.47, 127.80, 123.61, 122.16, 46.06, 35.78 (dd, J = 59.0, 28.9 Hz), 26.22.

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):** δ 60.04.

**HRMS (ESI, m/z):** Calculated for C<sub>22</sub>H<sub>29</sub>BrN<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup>450.1119, found 450.1192.

**30. 1-(4-bromophenyl)-3-(di-*p*-tolylphosphoryl)-2-(pyridin-2-yl)propan-1-one (4bf)**

A white solid, 189 mg, 73 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):** δ 8.25 (d, J = 4.2 Hz, 1H), 7.67 (d, J = 8.5 Hz, 2H), 7.47 (dd, J = 11.4, 8.0 Hz, 2H), 7.37 - 7.32 m, 3H), 7.28 (t, J = 7.6 Hz, 1H), 7.16 - 7.13 (m, 2H), 7.08 - 7.06 (m, 2H), 6.99 (dd, J = 8.0, 2.6 Hz, 2H), 6.85 (dd, J = 7.6, 5.0 Hz, 1H), 5.30 - 5.24 (m, 1H), 3.32 - 3.24 (m, 1H), 2.91 - 2.83 (m, 1H), 2.22 (d, J = 5.5 Hz, 6H).

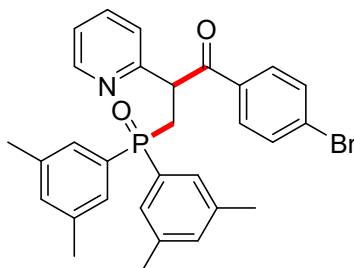
**<sup>13</sup>C NMR (101 MHz, Chloroform-d):** δ 196.09 (d, J = 8.1 Hz), 157.65, 149.73, 142.17, 141.68, 136.74, 134.56, 131.62, 130.70 (d, J = 9.9 Hz), 130.47, 129.20 (d, J = 22.4 Hz), 129.08 (d, J = 22.3 Hz), 128.14, 123.97, 122.03, 49.22, 32.08 (d, J = 71.9 Hz), 21.47 (d, J = 5.4 Hz).

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):** δ 30.73.

HRMS (ESI, m/z): Calculated for C<sub>28</sub>H<sub>25</sub>Br<sub>1</sub>N<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup> 518.0806, found 518.0880.

**31. 3-(bis(3,5-dimethylphenyl)phosphoryl)-1-(4-bromophenyl)-2-(pyridin-2-yl)propan-1-one (4bg)**

A white solid, 170.6 mg, 62 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



<sup>1</sup>H NMR (400 MHz, Chloroform-d): δ 8.29 (d, J = 4.9 Hz, 1H), 7.72 (d, J = 8.2 Hz, 2H), 7.36 (d, J = 8.2 Hz, 2H), 7.23 (d, J = 11.6 Hz, 3H), 7.18 (d, J = 7.5 Hz, 1H), 7.09 (d, J = 11.9 Hz, 2H), 6.93 (s, 1H), 6.87 (s, 1H), 6.82 (t, J = 6.2 Hz, 1H), 5.34 - 5.32 (m, 1H), 3.31 - 3.23 (m, 1H), 3.01 - 2.93 (m, 1H), 2.15 (d, J = 14.0 Hz, 12H).

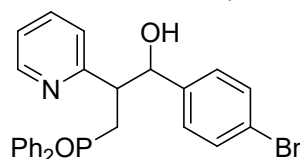
<sup>13</sup>C NMR (101 MHz, Chloroform-d): δ 195.70 (d, J = 8.6 Hz), 157.31 (d, J = 6.0 Hz), 149.18, 137.76 (d, J = 34.6 Hz), 137.64 (d, J = 34.6 Hz), 136.17, 134.26, 133.05 (d, J = 2.9 Hz), 132.88 (d, J = 99.2 Hz), 131.79 (d, J = 99.6 Hz), 132.76 (d, J = 2.9 Hz), 132.33 (d, J = 10.0 Hz), 131.30, 130.1, 127.88 (dd, J = 9.5 Hz), 127.82 (dd, J = 9.7 Hz), 123.74, 121.82, 48.84, 31.54 (d, J = 71.4 Hz), 20.87 (d, J = 5.8 Hz).

<sup>31</sup>P NMR (162 MHz, Chloroform-d): δ 30.53.

HRMS (ESI, m/z): Calculated for C<sub>30</sub>H<sub>29</sub>Br<sub>1</sub>N<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup> 546.1119, found 546.1193.

**32. (3-(4-bromophenyl)-3-hydroxy-2-(pyridin-2-yl)propyl)diphenylphosphine oxide (4ac-1)**

A white solid, 90 mg, 80 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



<sup>1</sup>H NMR (400 MHz, Chloroform-d): δ 8.35 (s, 1H), 7.80 (t, J = 9.1 Hz, 2H), 7.61 - 7.49 (m, 5H), 7.43 - 7.33 (m, 2H), 7.27 (d, J = 8.0 Hz, 3H), 7.20 (t, J = 8.8 Hz, 1H), 6.93 (d, J = 8.0 Hz, 3H), 6.68 (d, J = 7.8 Hz, 1H), 5.07 (s, 1H), 3.79 (t, J = 9.4 Hz, 1H), 3.28 - 3.17 (m, 1H), 3.13 - 2.95 (m, 1H), 2.17 (s, 1H).

<sup>13</sup>C NMR (101 MHz, Chloroform-d): δ 159.54 (d, J = 4.2 Hz), 147.99, 142.17, 136.65, 136.51, 133.76 (d, J = 100.1 Hz), 131.88 (d, J = 98.9 Hz), 131.79 (d, J = 2.5 Hz), 131.40, 131.12 (d, J = 2.6 Hz), 130.48 (d, J = 27.3 Hz), 130.39 (d, J = 27.2 Hz), 128.67 (d, J = 11.7 Hz), 128.14 (d, J = 11.8 Hz), 127.21, 125.35, 122.10, 120.52, 45.62 (d, J = 2.7 Hz), 33.25 (d, J = 71.5 Hz).

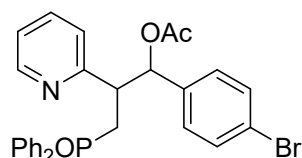
<sup>31</sup>P NMR (162 MHz, Chloroform-d): δ 30.74.



**HRMS (ESI, m/z):** Calculated for C<sub>26</sub>H<sub>23</sub>Br<sub>1</sub>N<sub>1</sub>O<sub>2</sub>P<sub>1</sub> (M+H)<sup>+</sup>492.0650, found 492.0736.

**33. 1-(4-bromophenyl)-3-(diphenylphosphoryl)-2-(pyridin-2-yl)propyl acetate (4ac-2)**

A white solid, 60 mg, 82 %, after purification by flash column chromatography (dichloromethane /ethyl acetate = 4:1 +1 % Et<sub>3</sub>N)



**<sup>1</sup>H NMR (400 MHz, Chloroform-d):** δ 8.25 (d, J = 4.2 Hz, 1H), 7.78 - 7.66 (m, 2H), 7.51 - 7.24 (m, 5H), 7.28 - 7.23 (m, 2H), 7.15 (d, J = 7.8 Hz, 3H), 7.08 (t, J = 7.7 Hz, 1H), 6.85 (d, J = 7.8 Hz, 3H), 6.61 (d, J = 7.8 Hz, 1H), 5.00 (s, 1H), 3.73 (d, J = 10.5 Hz, 1H), 3.19 - 3.11 (m, 1H), 2.98 (td, J = 14.7, 4.3 Hz, 1H), 1.22 (s, 3H).

**<sup>13</sup>C NMR (101 MHz, Chloroform-d):** δ 169.23, 158.32, 149.12, 137.01, 135.60, 133.58 (d, J = 100.0 Hz), 132.33 (d, J = 97.1 Hz), 131.85, 131.58 (d, J = 2.7 Hz), 130.94 (d, J = 2.8 Hz), 130.47 (d, J = 28.7 Hz), 130.37 (d, J = 28.4 Hz), 129.66, 128.59 (d, J = 11.7 Hz), 127.97 (d, J = 11.9 Hz), 125.72, 122.63, 121.99, 78.45 (d, J = 16.4 Hz), 45.64 (d, J = 2.2 Hz), 30.19 (d, J = 72.2 Hz), 20.60.

**<sup>31</sup>P NMR (162 MHz, Chloroform-d):** δ 31.15.

**HRMS (ESI, m/z):** Calculated for C<sub>28</sub>H<sub>25</sub>Br<sub>1</sub>N<sub>1</sub>O<sub>3</sub>P<sub>1</sub> (M+H)<sup>+</sup> 534.0755, found 534.0837.

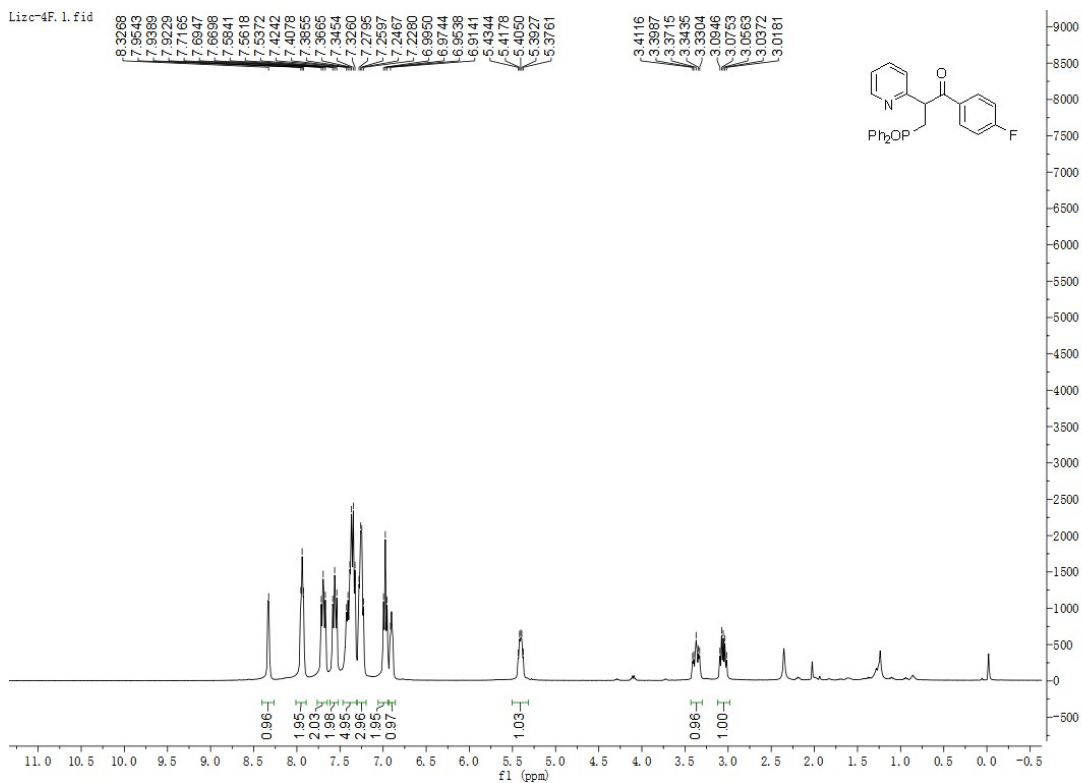
## 10. References

1. B. Zhou, Y. Hu and C. Y. Wang, Manganese-Catalyzed Direct Nucleophilic C(sp<sup>2</sup>)-H Addition to Aldehydes and Nitriles, *Angew. Chem. Int. Ed.*, 2015, **54**, 13659-13663.
2. M. Chaitanya and P. Anbarasan, Rhodium Catalyzed Cyanation of C(sp<sup>2</sup>)-H Bond of Alkenes, *Org. Lett.*, 2015, **17**, 3766–3769.
3. H. Ruan, L-G. Meng, H. Xu, Y. Liang and L. Wang, Additive-free coupling of bromoalkynes with secondary phosphine oxides to generate alkynylphosphine oxides in acetic anhydride, *Org. Biomol. Chem.*, 2020, **18**, 1087–1090.

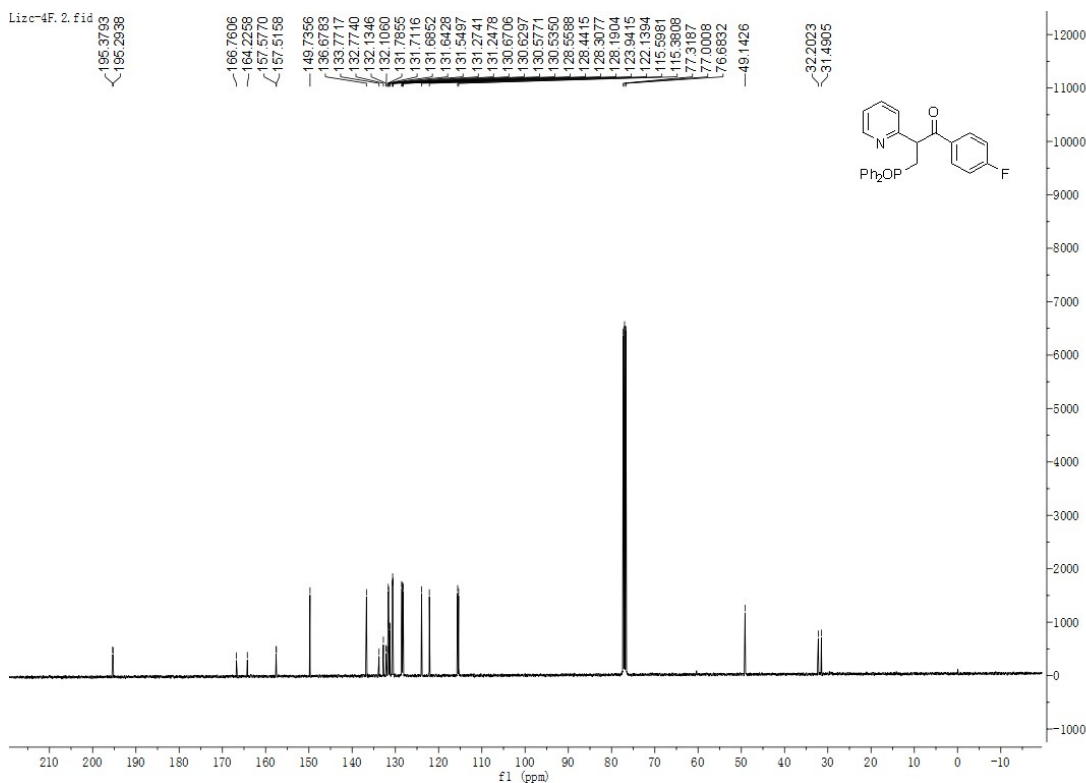
## 11. Copies of the NMR spectra.

### 1. 3-(diphenylphosphoryl)-1-(4-fluorophenyl)-2-(pyridin-2-yl)propan-1-one (4aa)

<sup>1</sup>H NMR

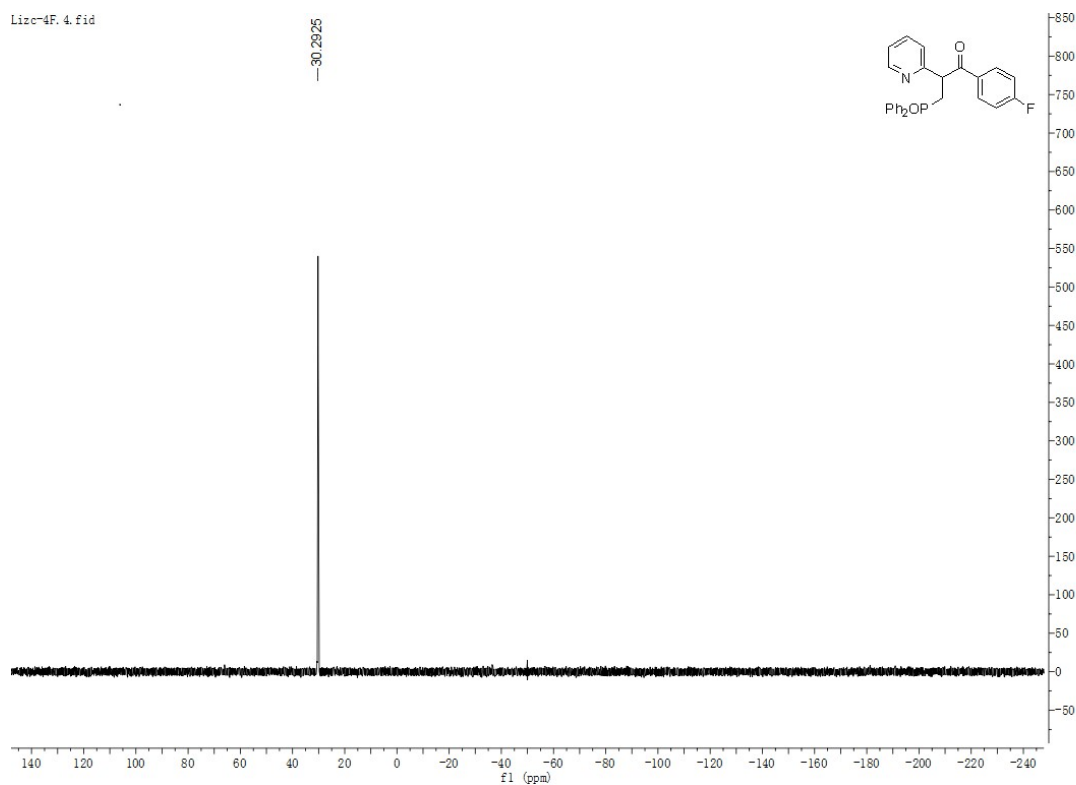


<sup>13</sup>C NMR



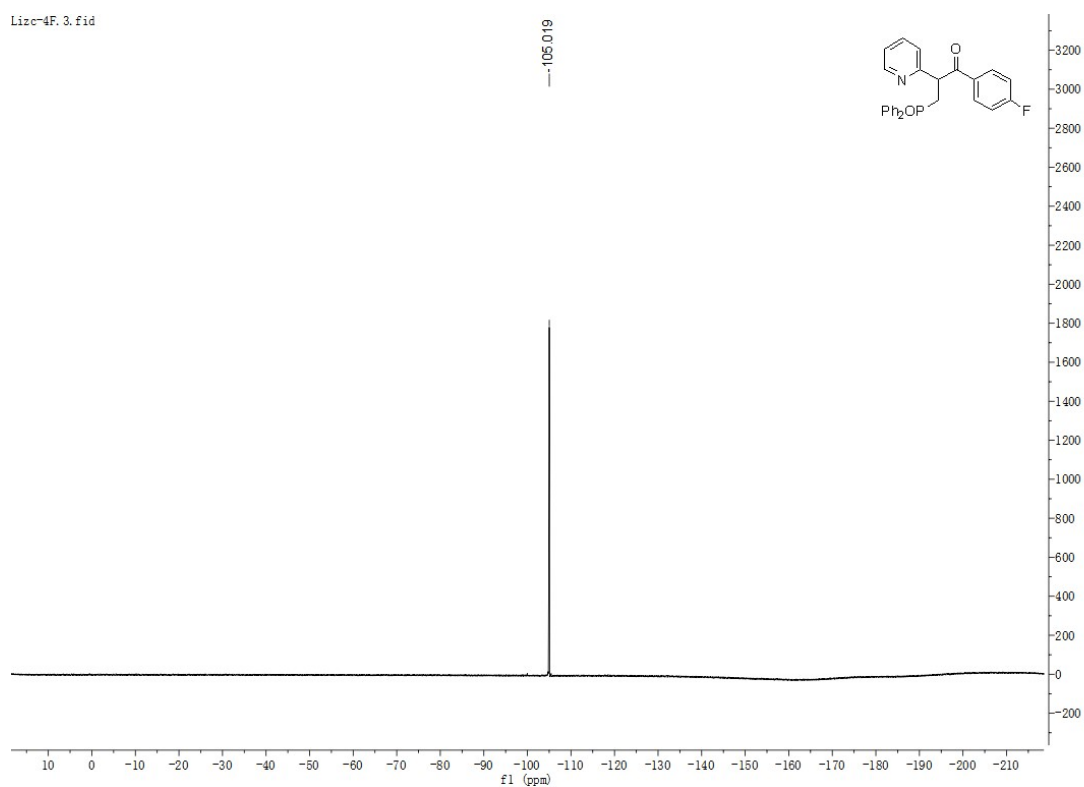
### <sup>31</sup>P NMR

Lize-4F. 4.fid



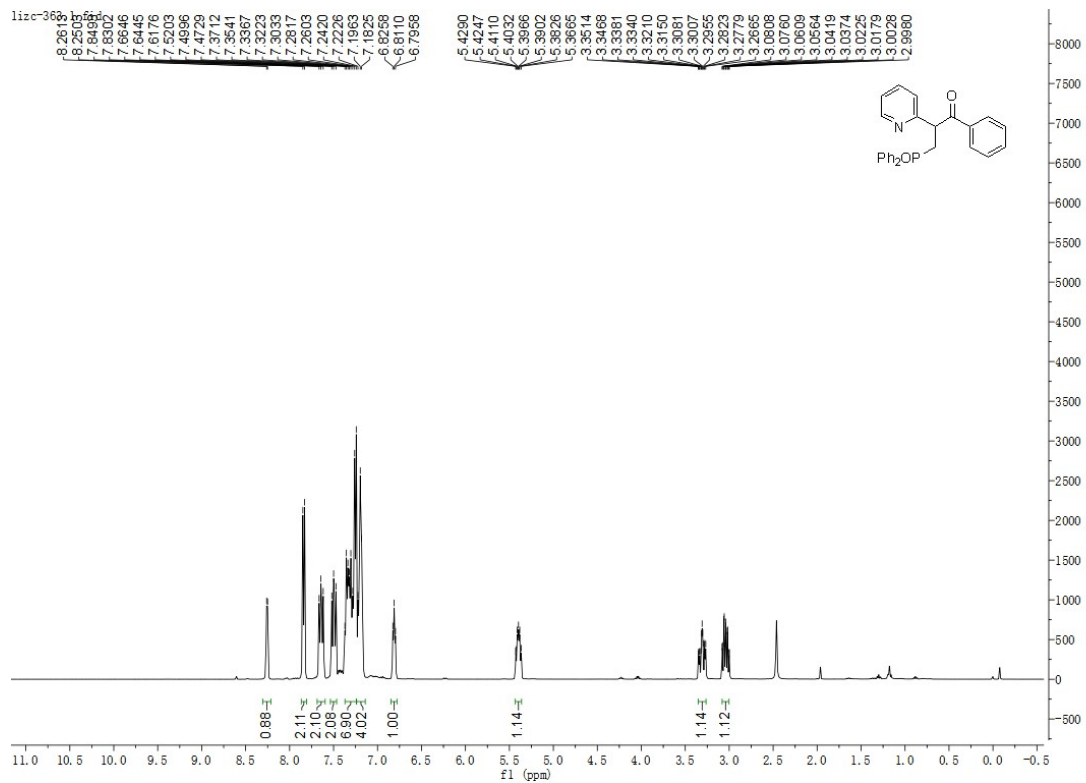
### <sup>19</sup>F NMR

Lize-4F. 3.fid

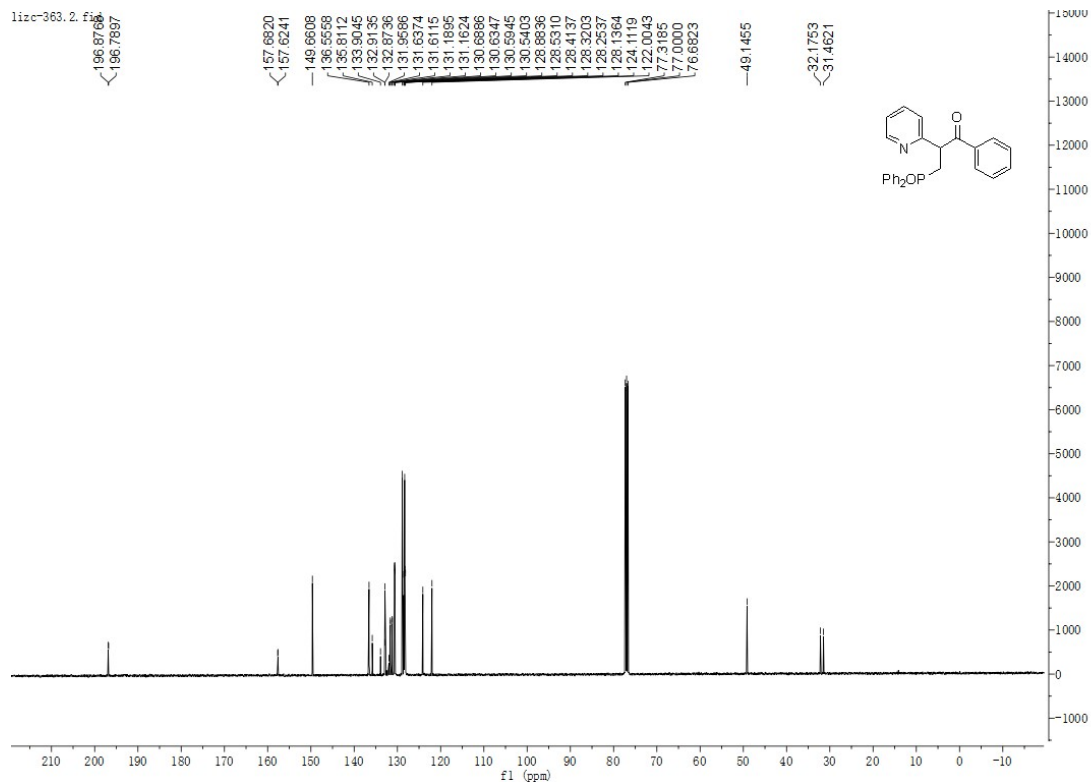


## 2. 3-(diphenylphosphoryl)-1-phenyl-2-(pyridin-2-yl)propan-1-one (4ab)

### <sup>1</sup>H NMR

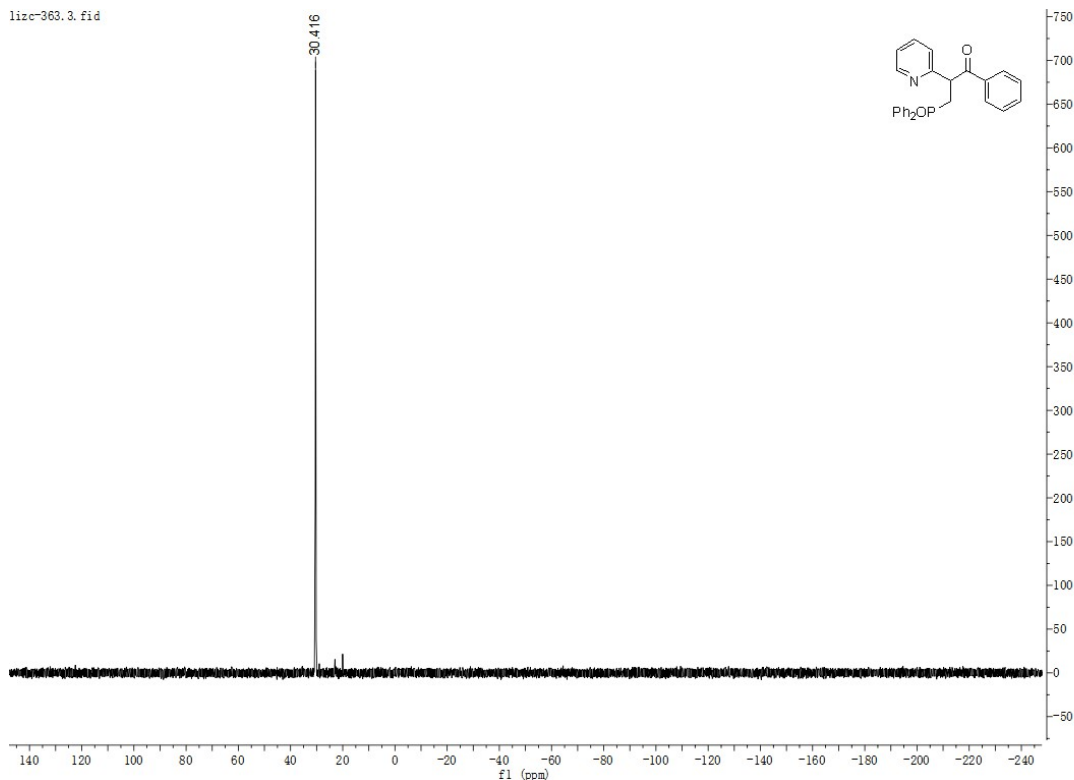


### <sup>13</sup>C NMR



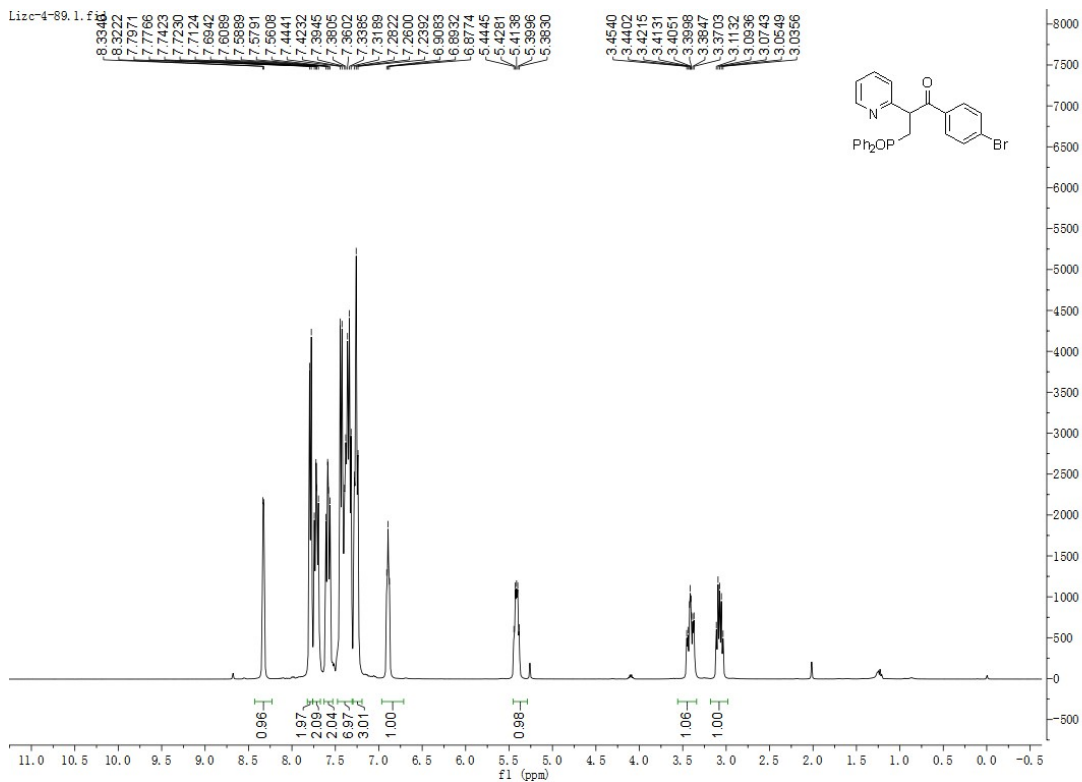
### <sup>31</sup>P NMR

lizc-363.3.fid

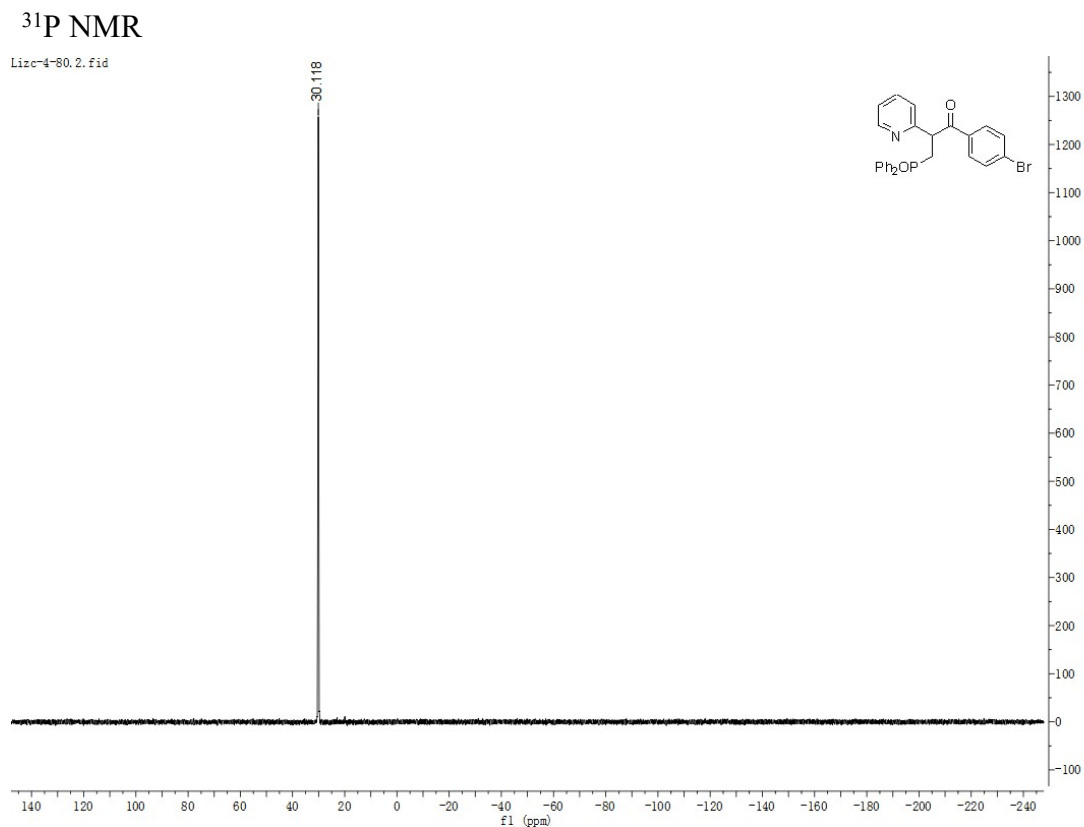
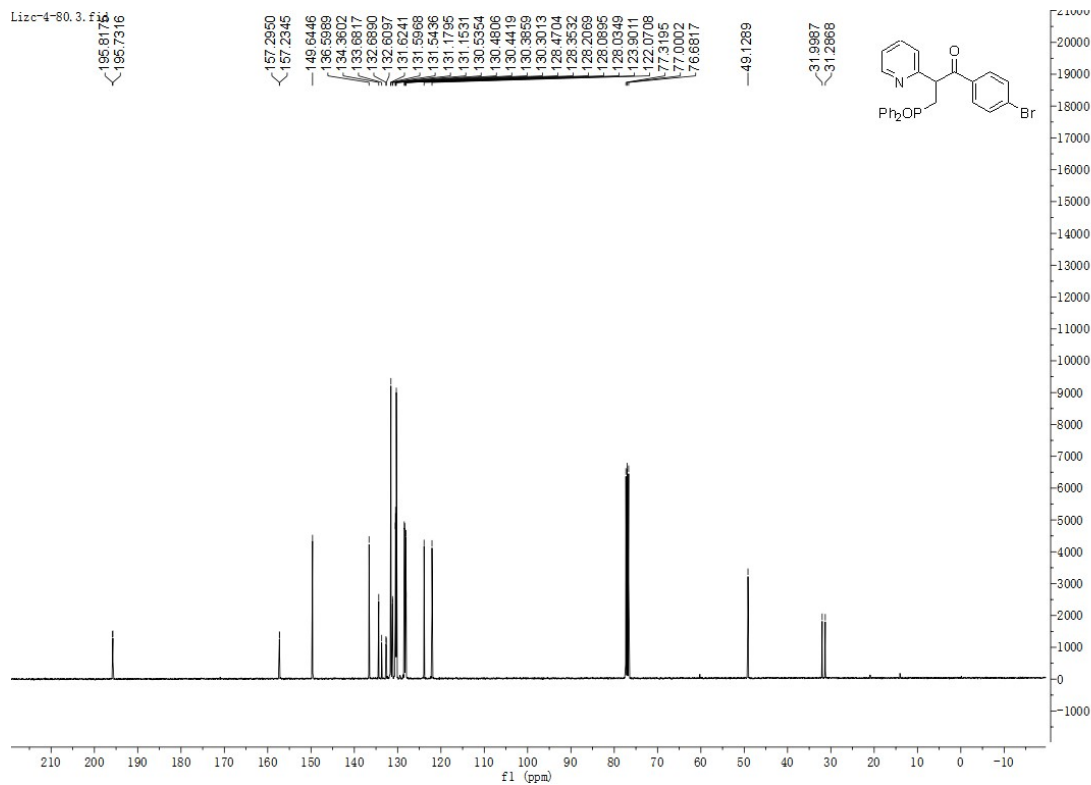


### 3. 1-(4-bromophenyl)-3-(diphenylphosphoryl)-2-(pyridin-2-yl)propan-1-one (4ac)

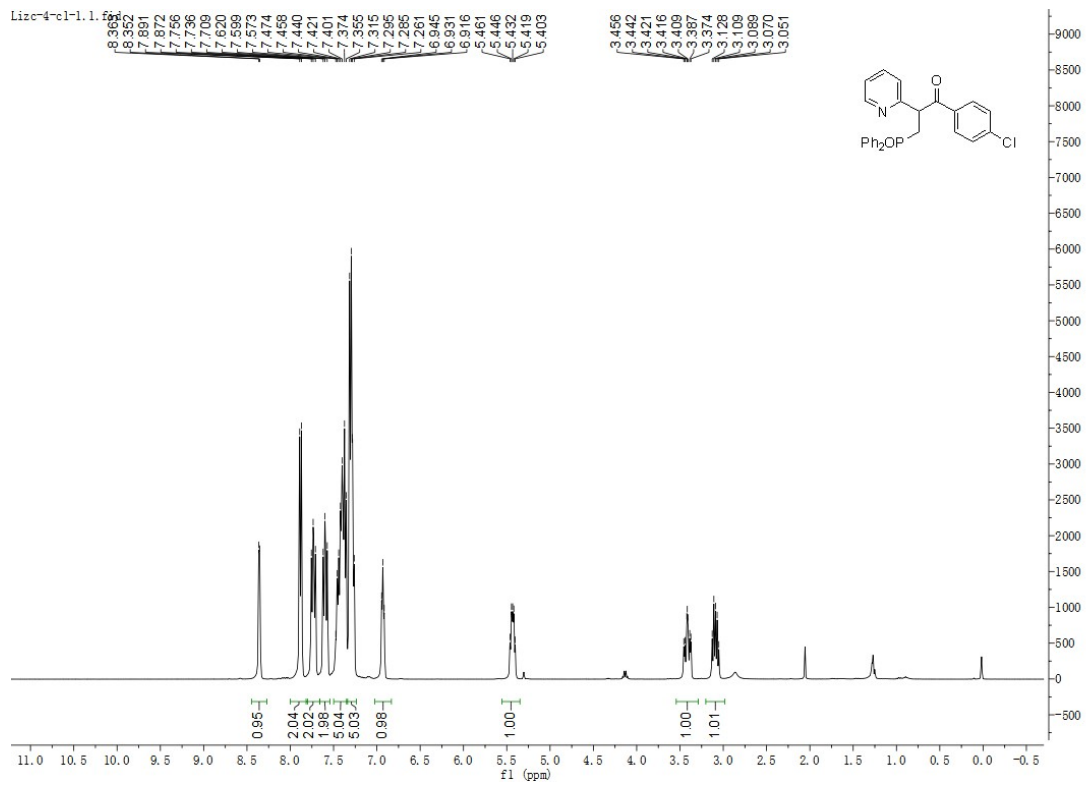
<sup>1</sup>H NMR



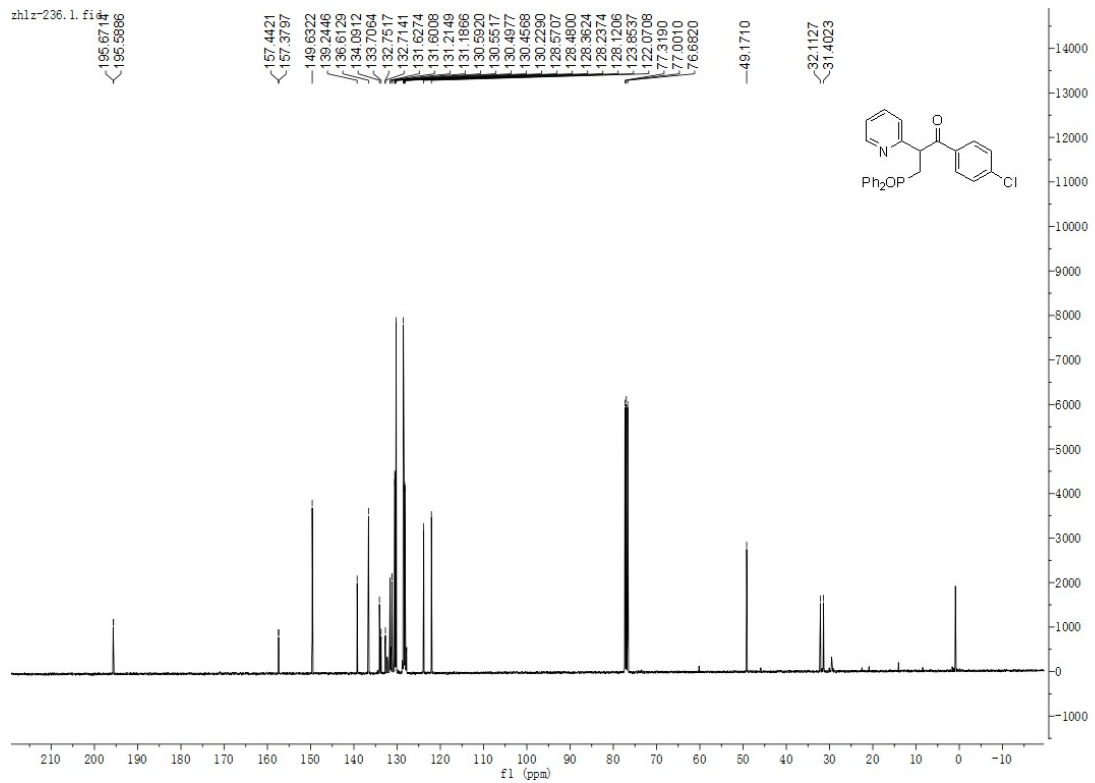
<sup>13</sup>C NMR



**4. 1-(4-chlorophenyl)-3-(diphenylphosphoryl)-2-(pyridin-2-yl)propan-1-one (4ad)**  
<sup>1</sup>H NMR

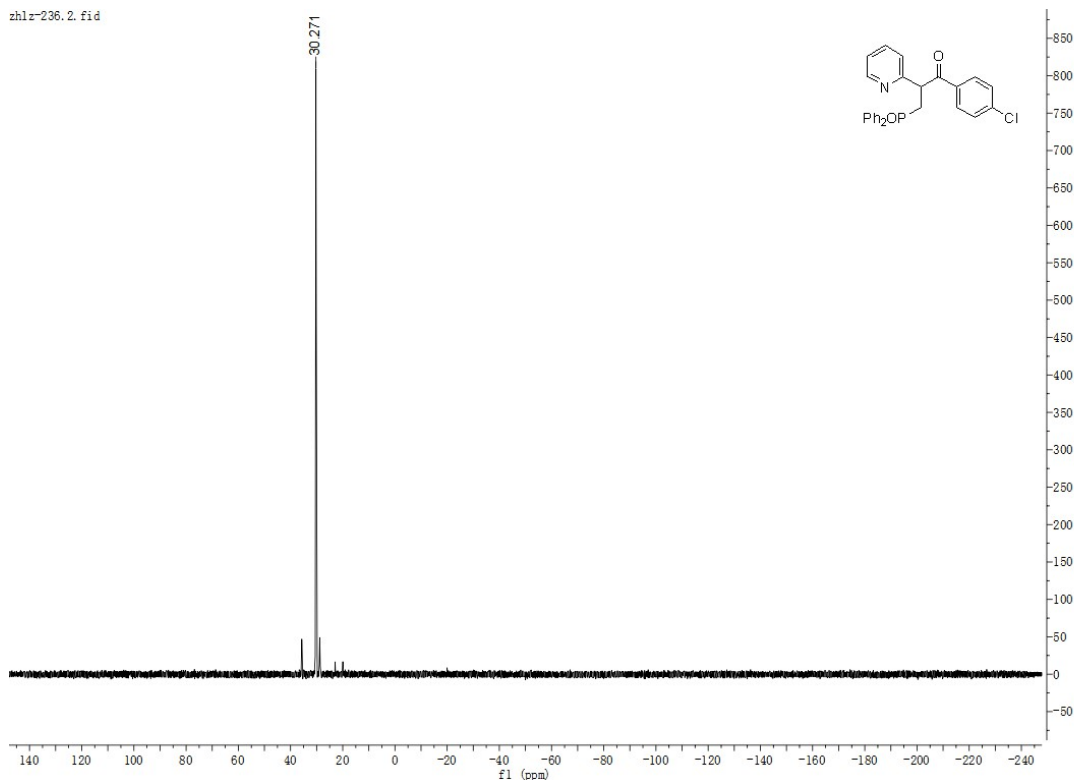


### <sup>13</sup>C NMR



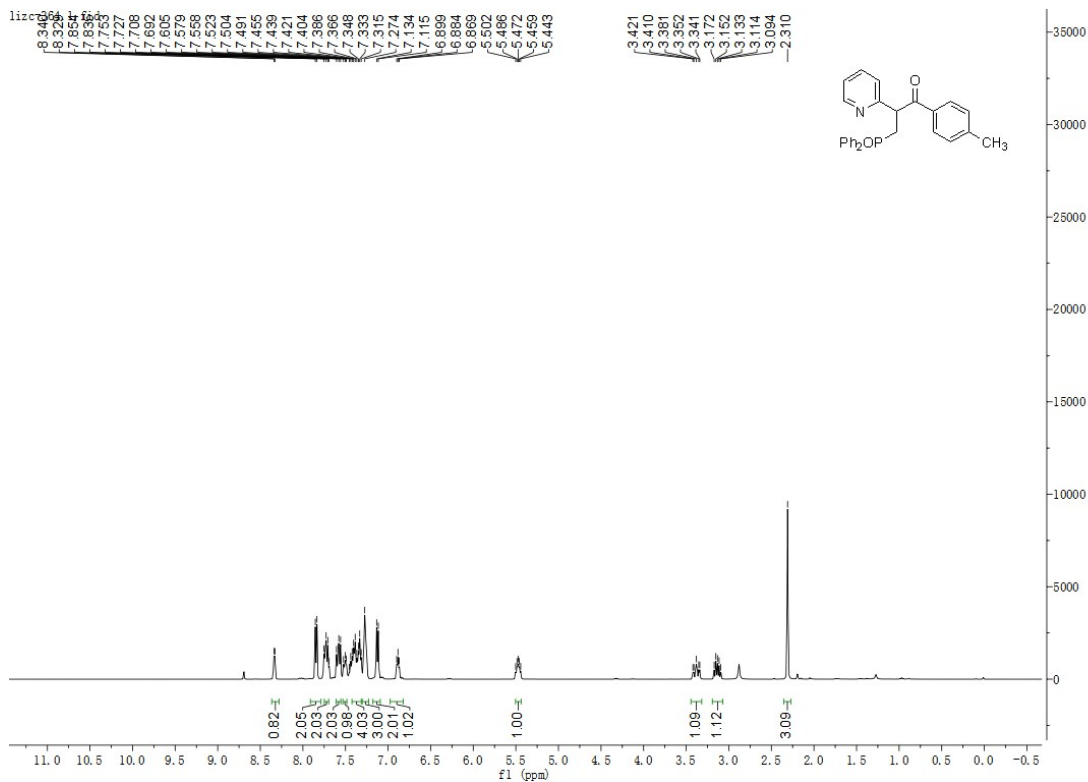
### <sup>31</sup>P NMR

zhlz-236.2.fid



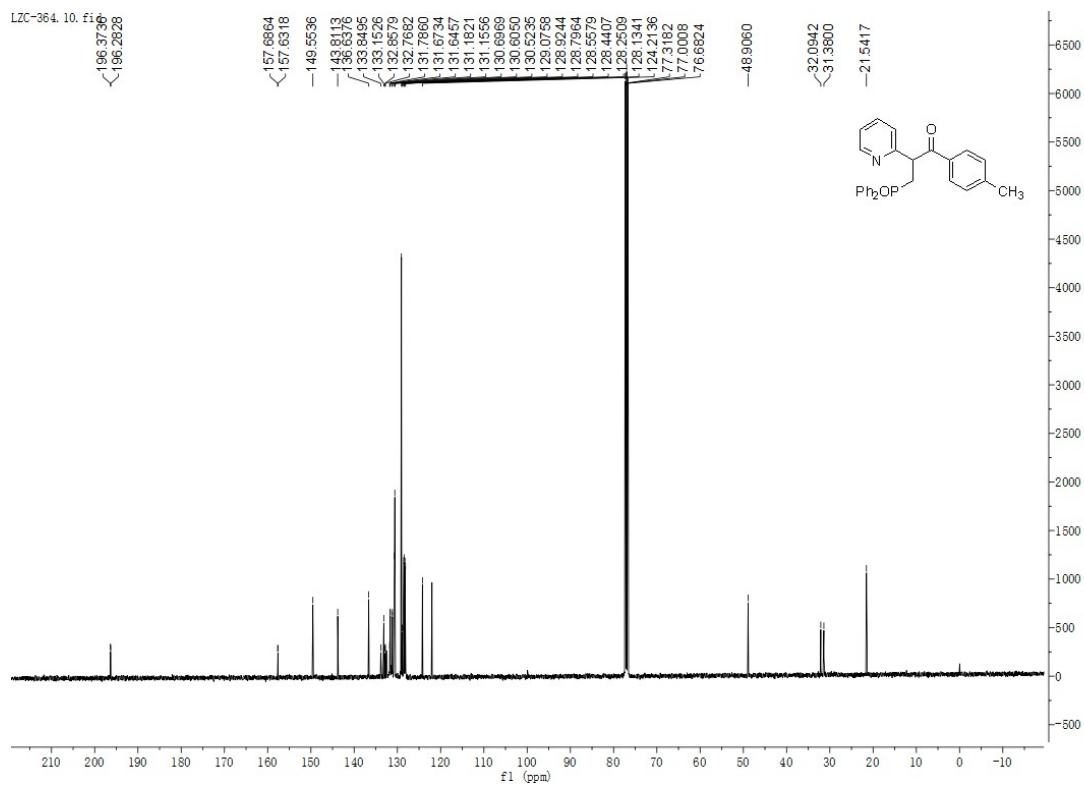
### 5. 3-(diphenylphosphoryl)-2-(pyridin-2-yl)-1-(p-tolyl)propan-1-one (4ae)

<sup>1</sup>H NMR

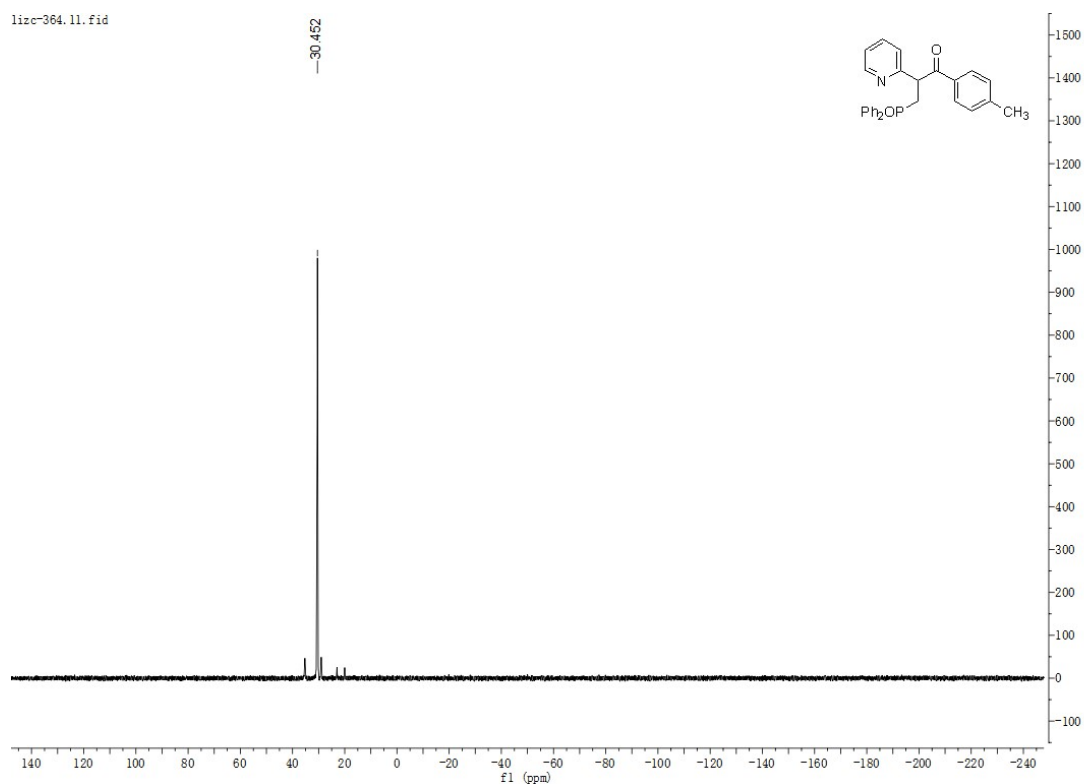


<sup>13</sup>C NMR





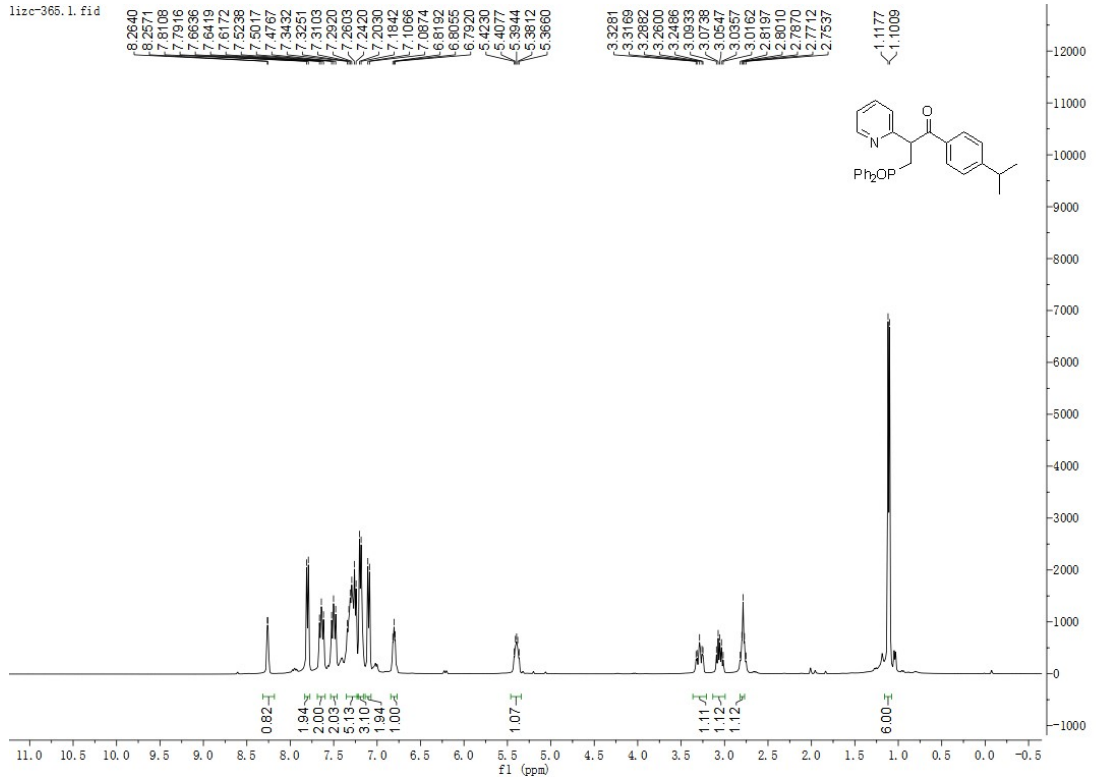
### <sup>31</sup>P NMR



## 6. 3-(diphenylphosphoryl)-1-(4-isopropylphenyl)-2-(pyridin-2-yl)propan-1-one (4af)

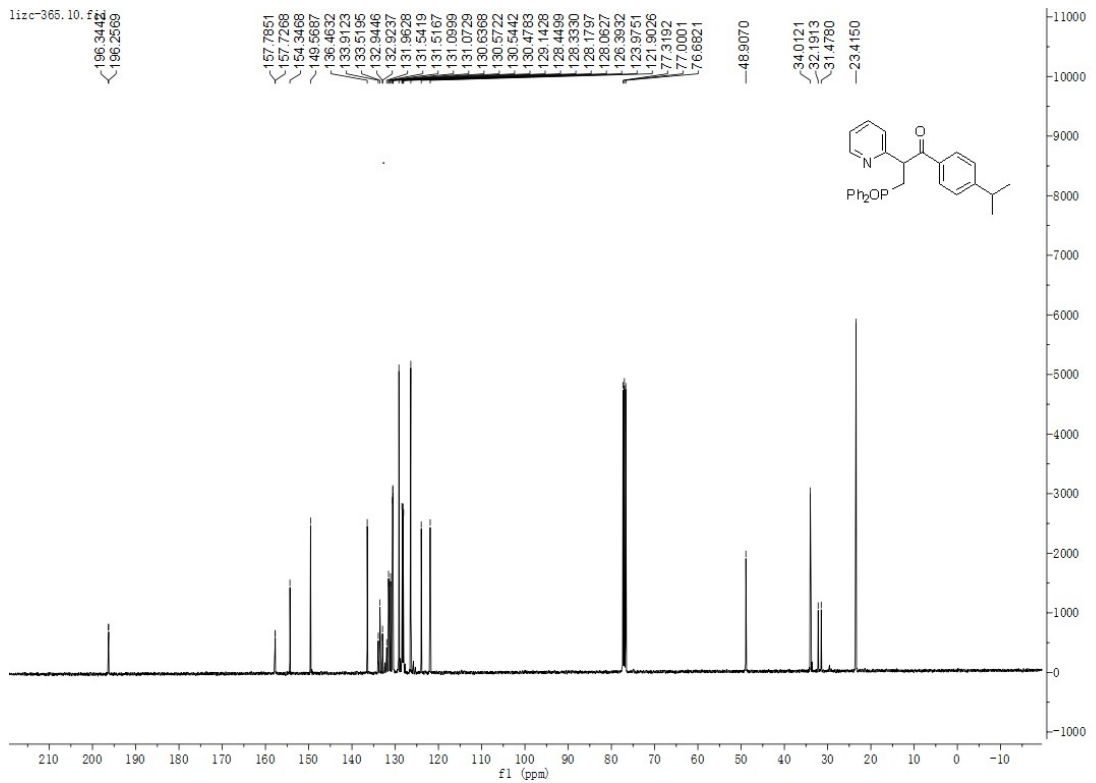
### <sup>1</sup>H NMR

lizc-365.1.fid



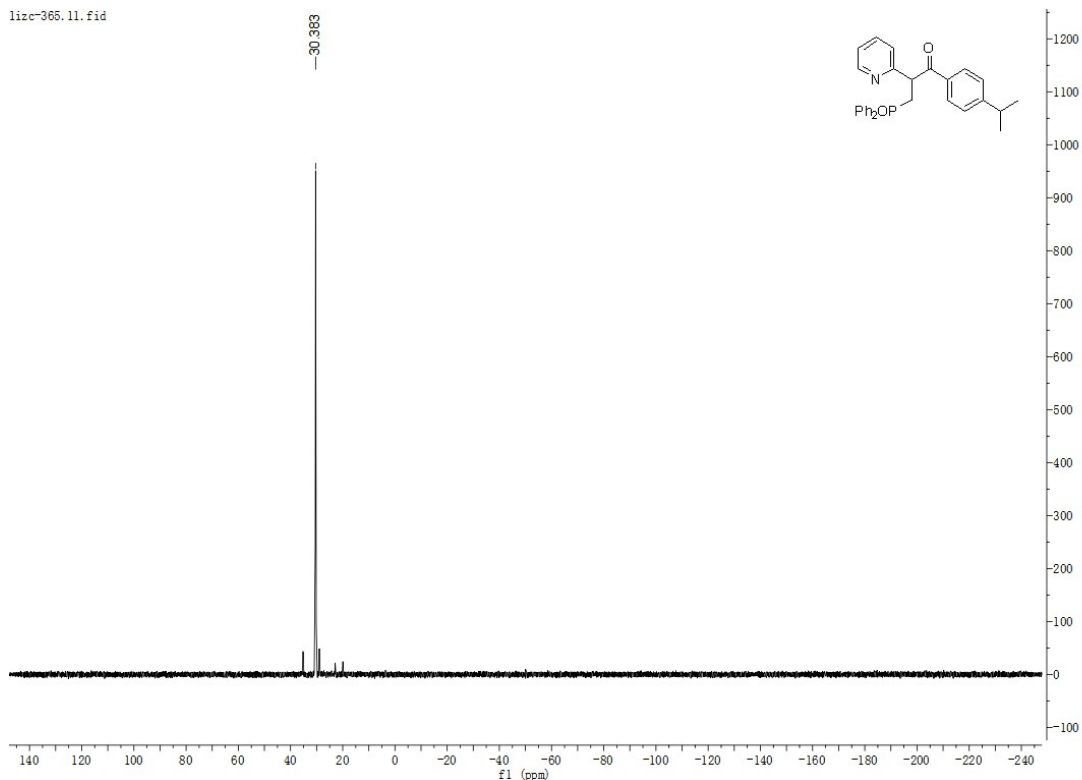
### <sup>13</sup>C NMR

lizc-365.10.fid



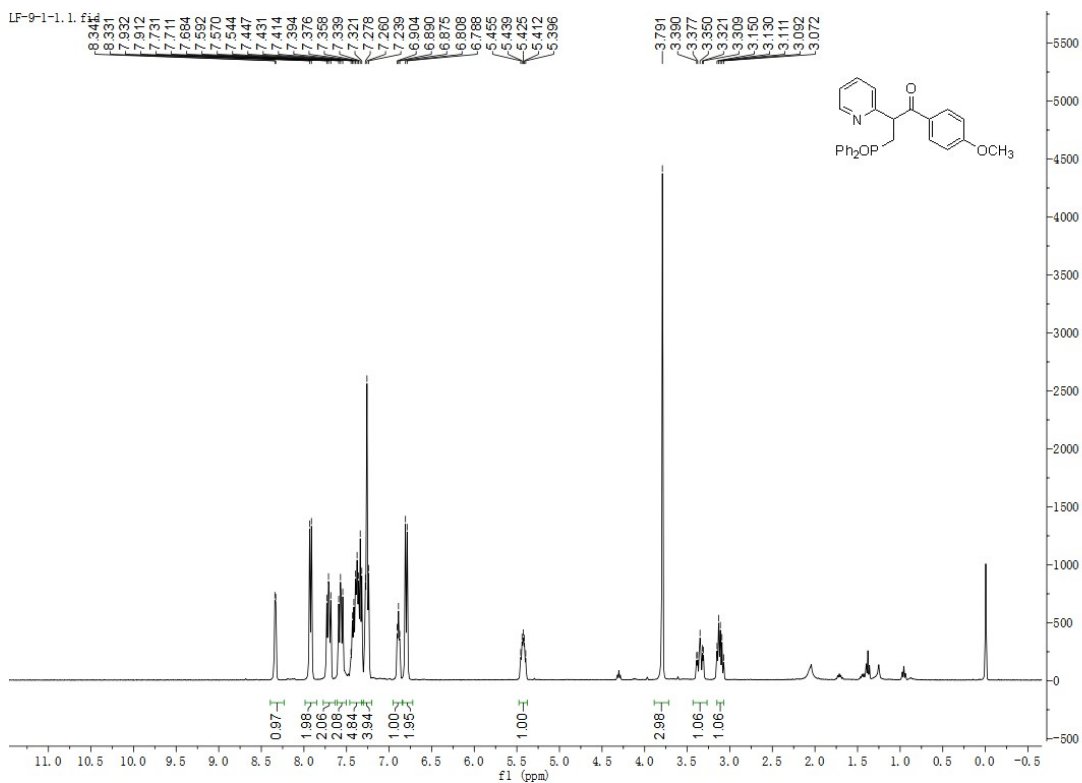
### <sup>31</sup>P NMR

lizc-365.11.fid

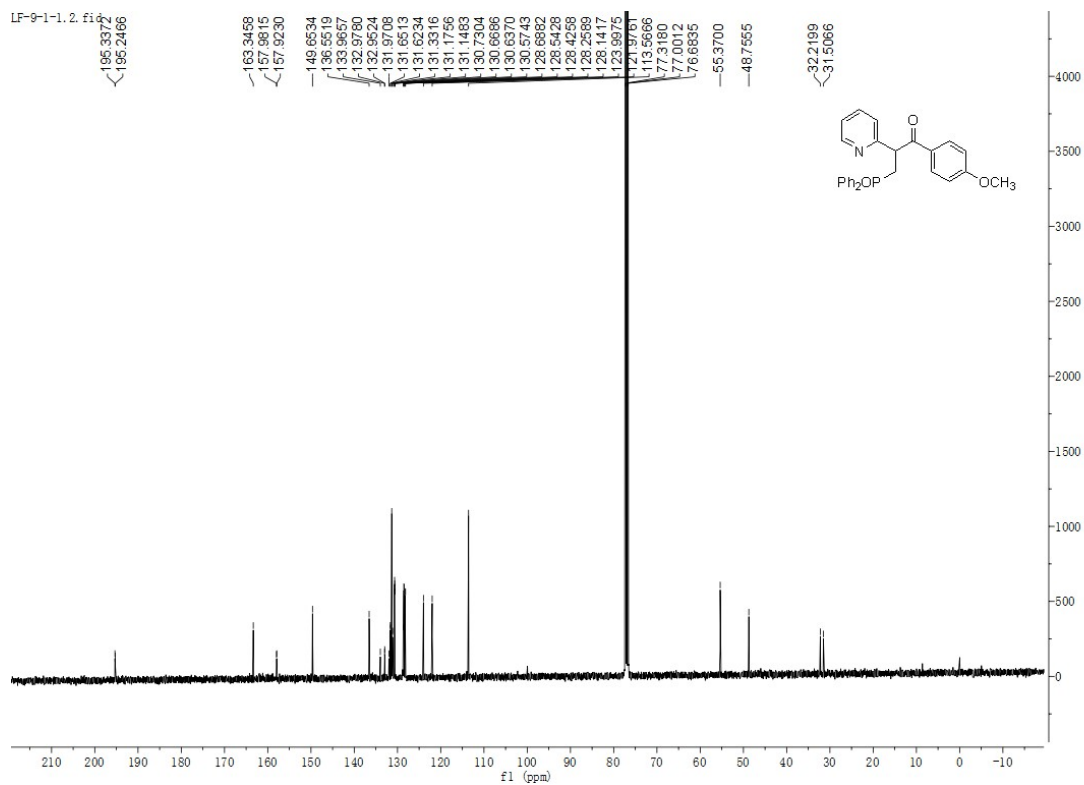


### 7. 3-(diphenylphosphoryl)-1-(4-methoxyphenyl)-2-(pyridin-2-yl)propan-1-one (4ag)

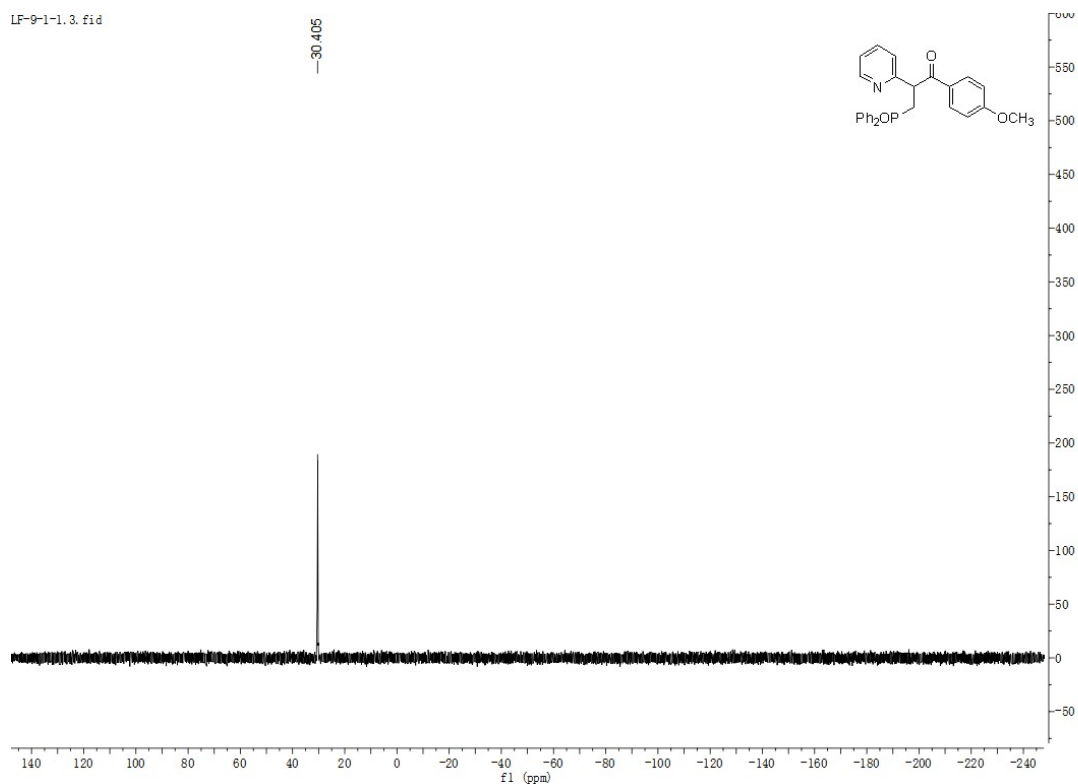
<sup>1</sup>H NMR



<sup>13</sup>C NMR

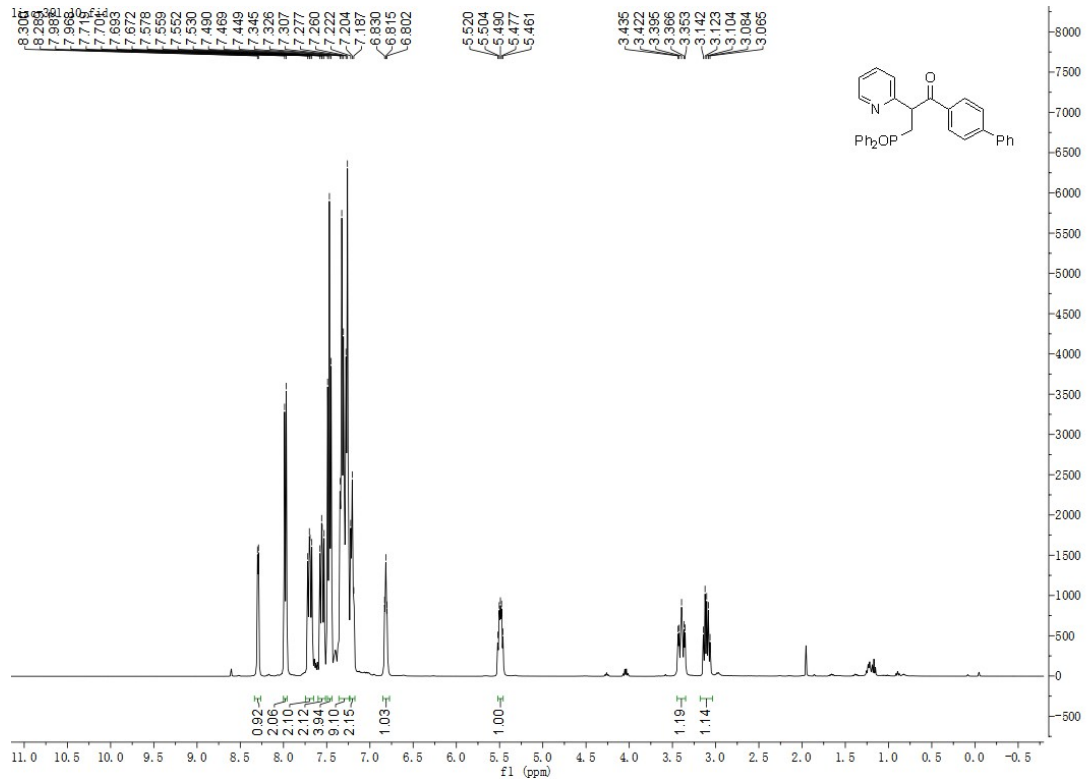


### <sup>31</sup>P NMR

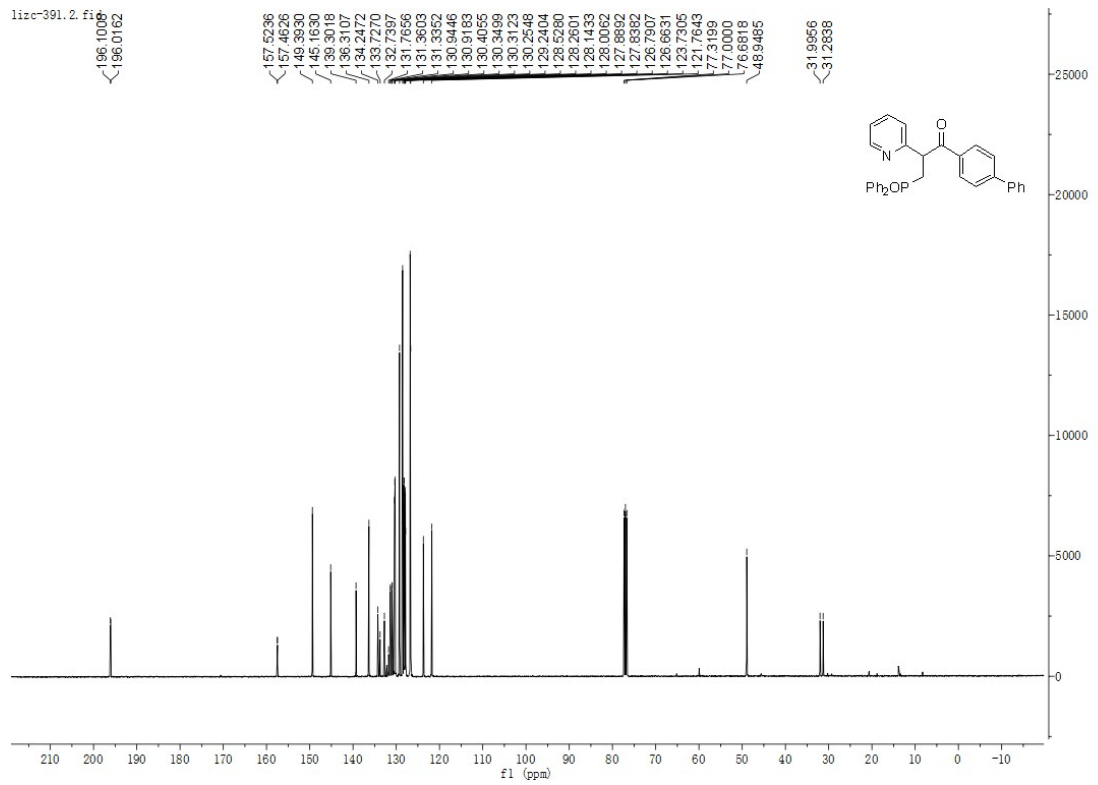


## 8. 1-([1,1'-biphenyl]-4-yl)-3-(diphenylphosphoryl)-2-(pyridin-2-yl)propan-1-one (4ah)

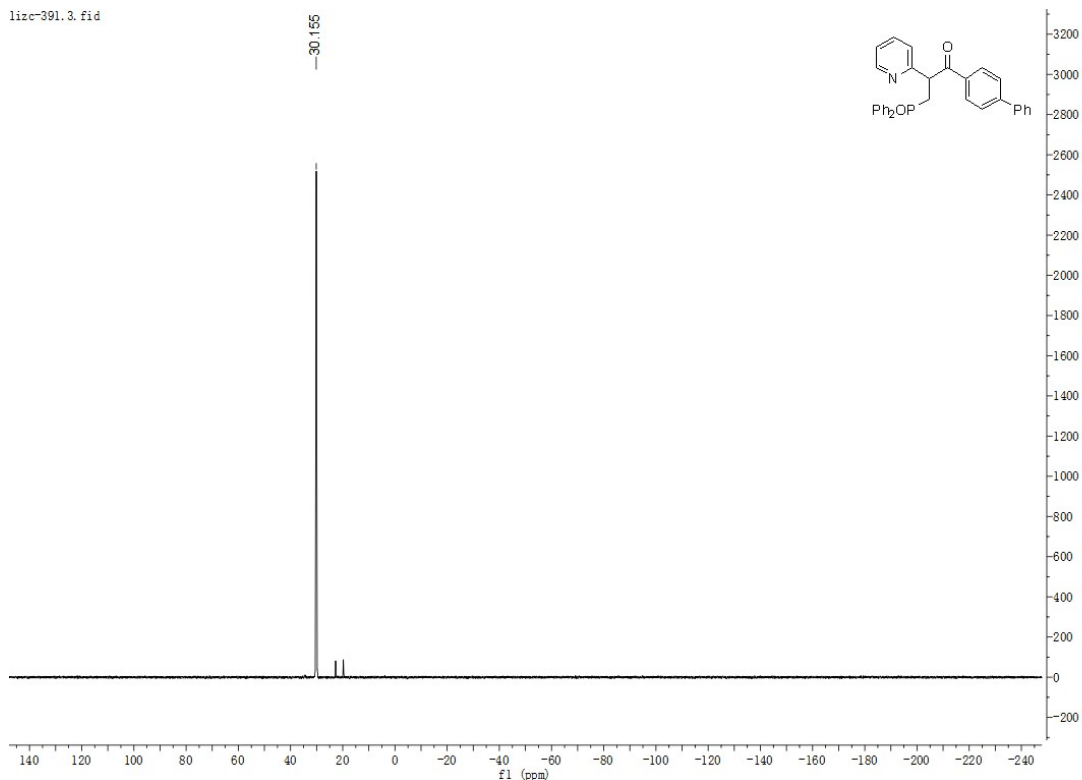
### <sup>1</sup>H NMR



### <sup>13</sup>C NMR

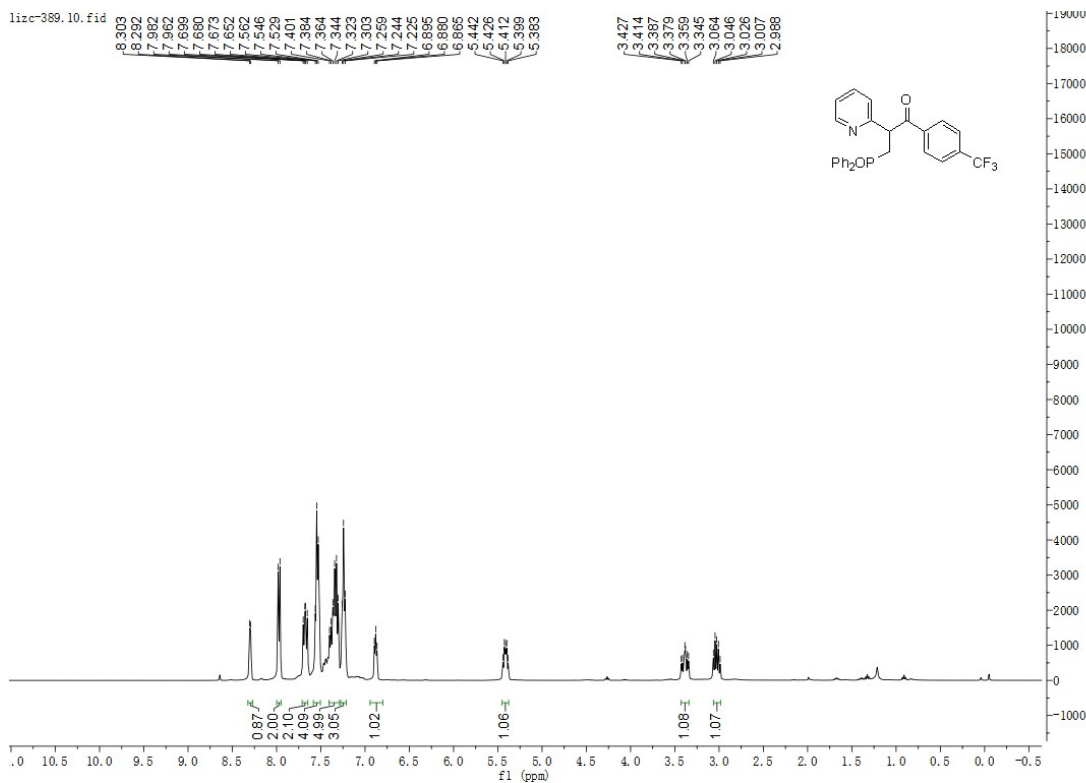


### <sup>31</sup>P NMR

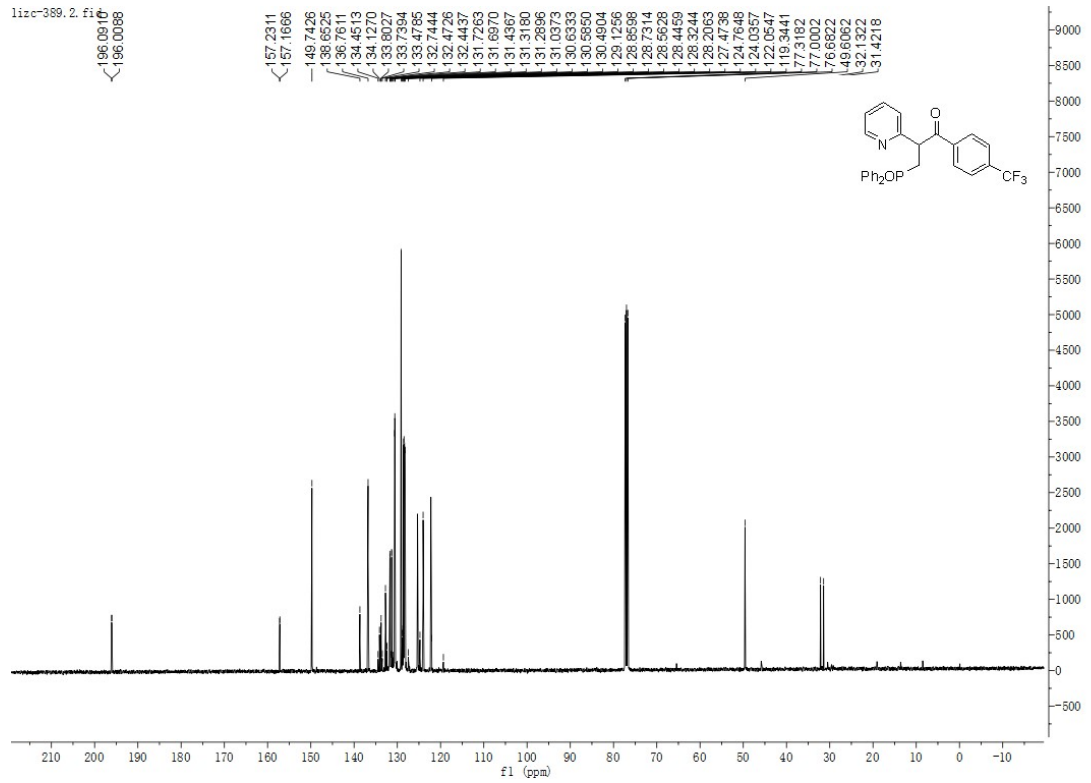


### 9. 3-(diphenylphosphoryl)-2-(pyridin-2-yl)-1-(4-(trifluoromethyl)phenyl)propan-1-one (4ai)

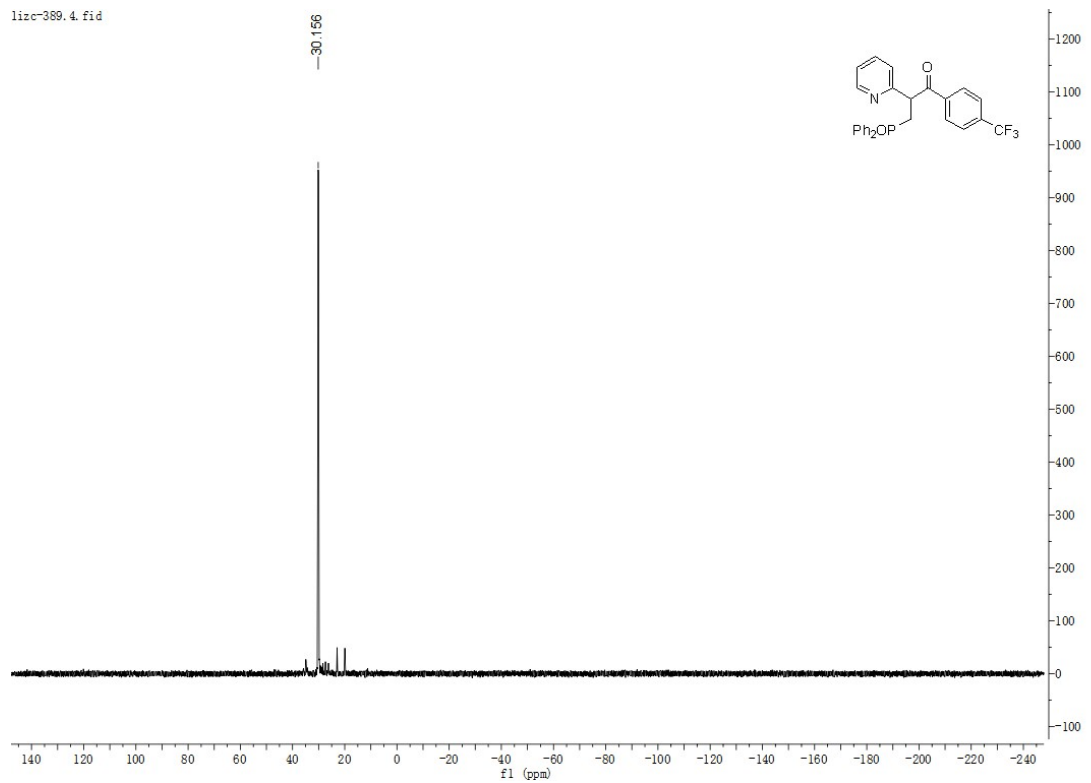
<sup>1</sup>H NMR



<sup>13</sup>C NMR

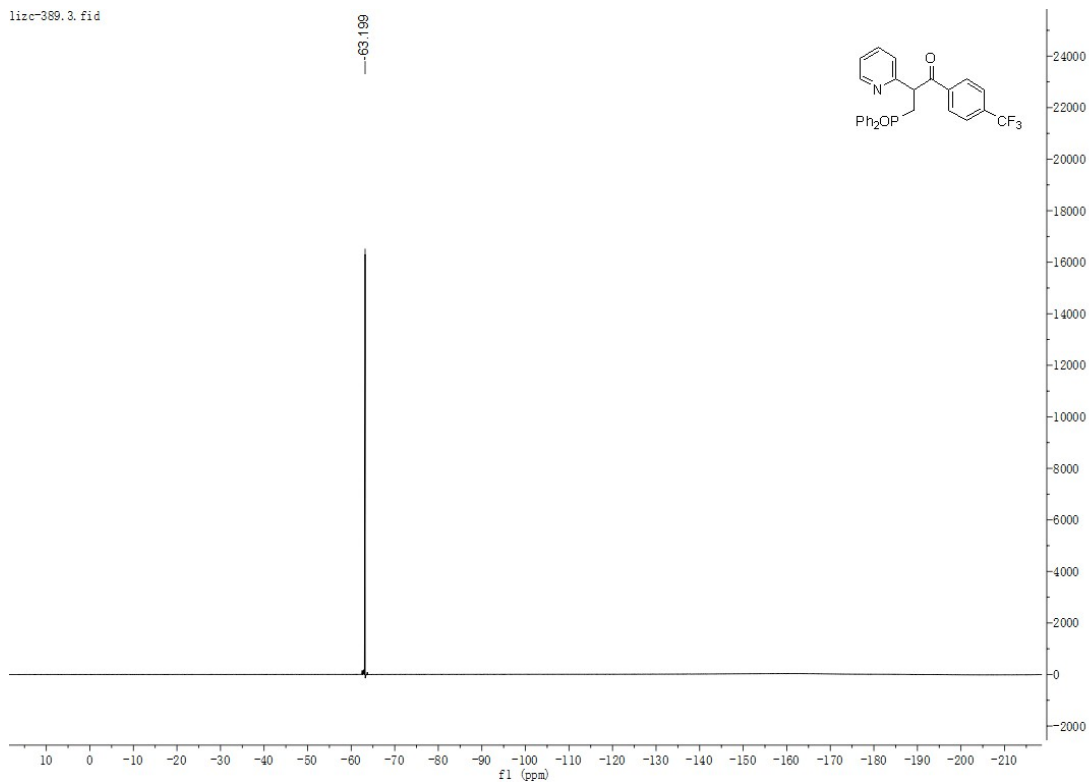


### <sup>31</sup>P NMR



### <sup>19</sup>F NMR

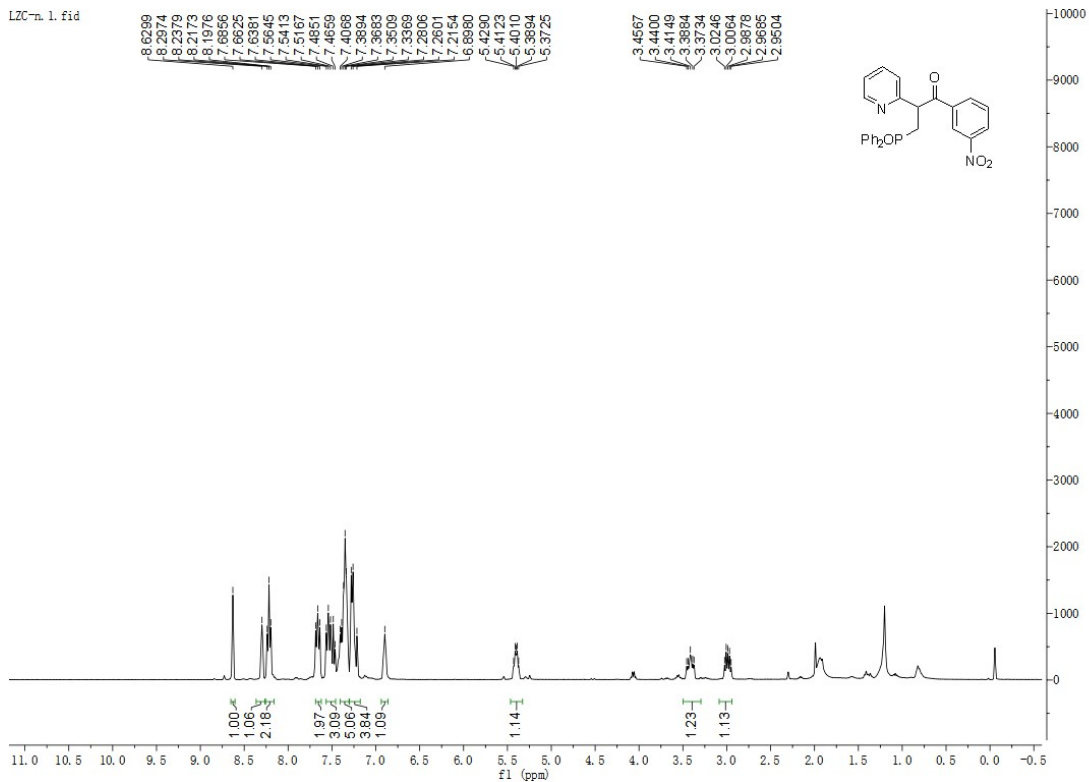
lzc-389.3.fid



### 10. 3-(diphenylphosphoryl)-1-(3-nitrophenyl)-2-(pyridin-2-yl)propan-1-one (4aj)

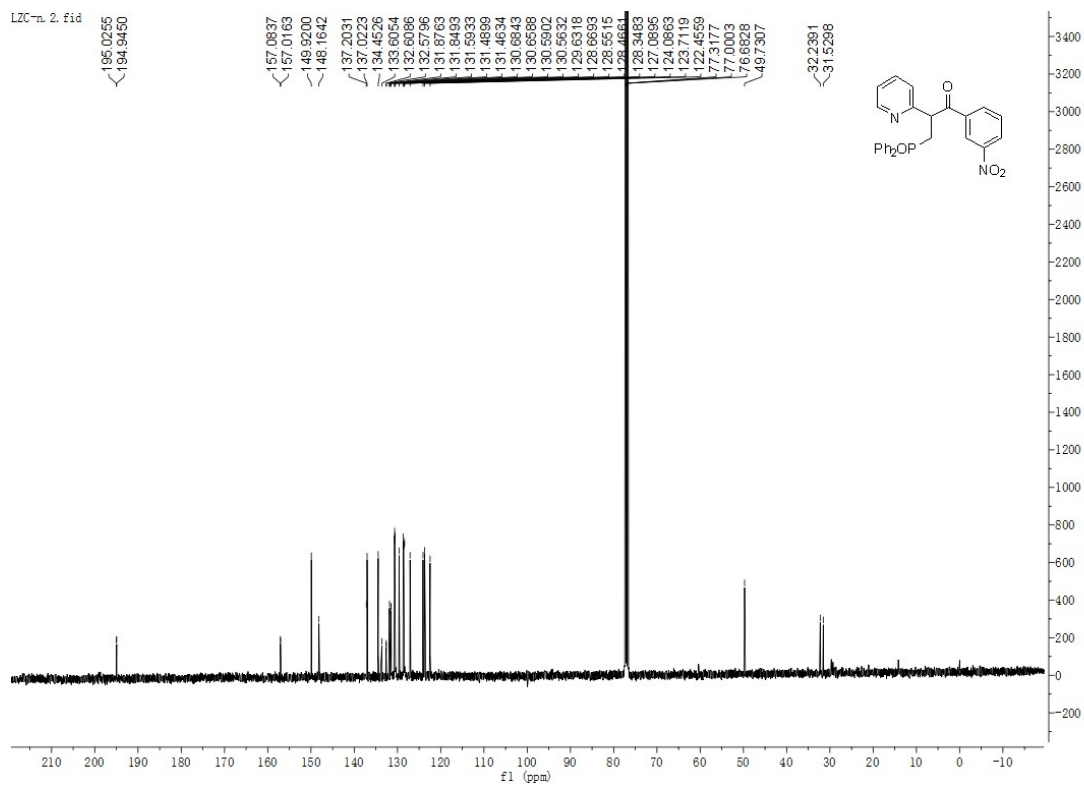
<sup>1</sup>H NMR

LZC-n.1.fid

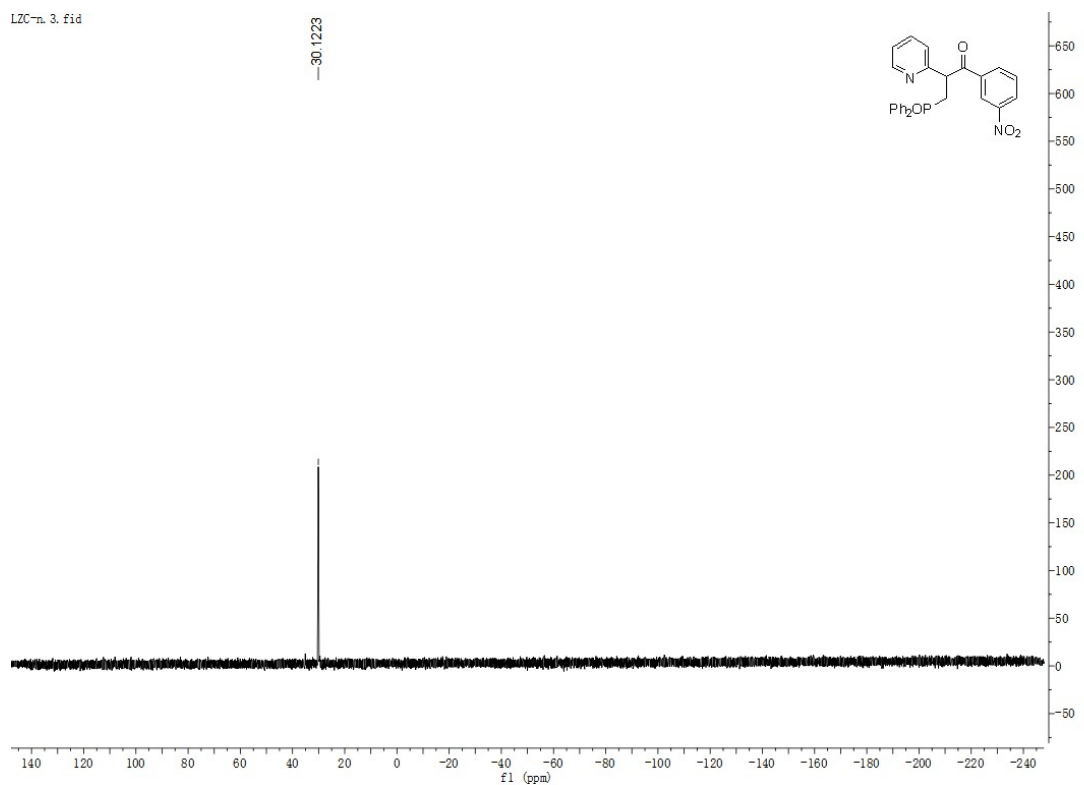


<sup>13</sup>C NMR



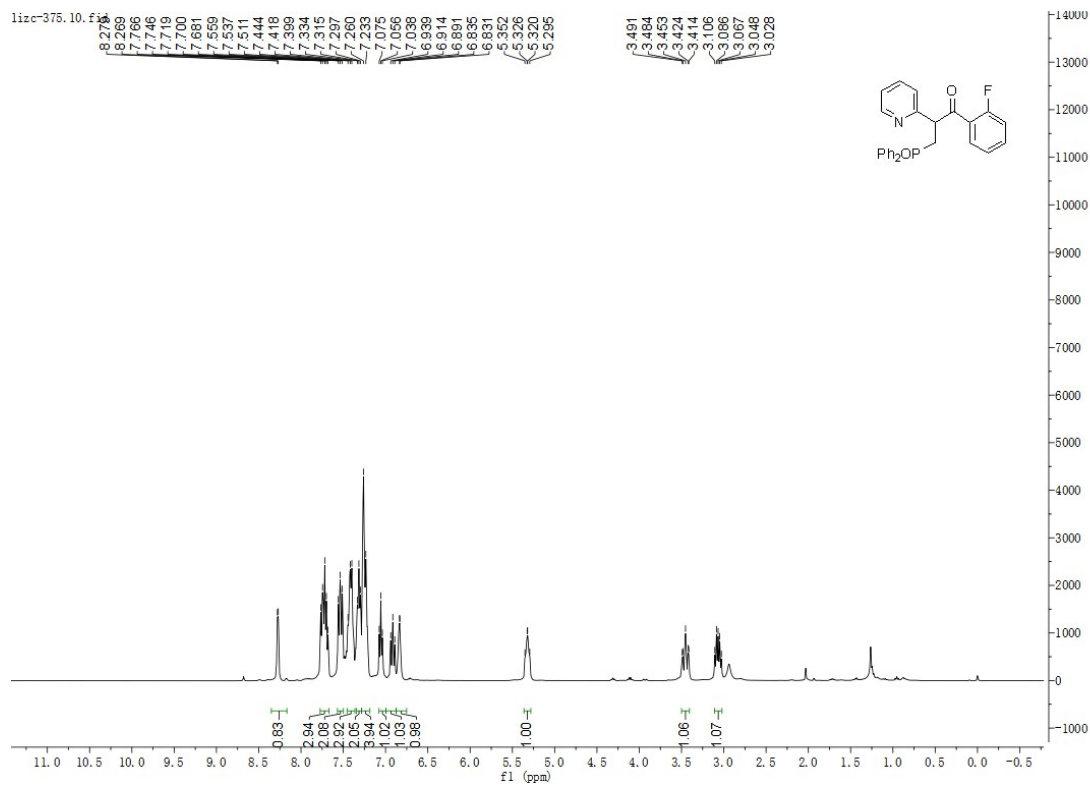


### <sup>31</sup>P NMR

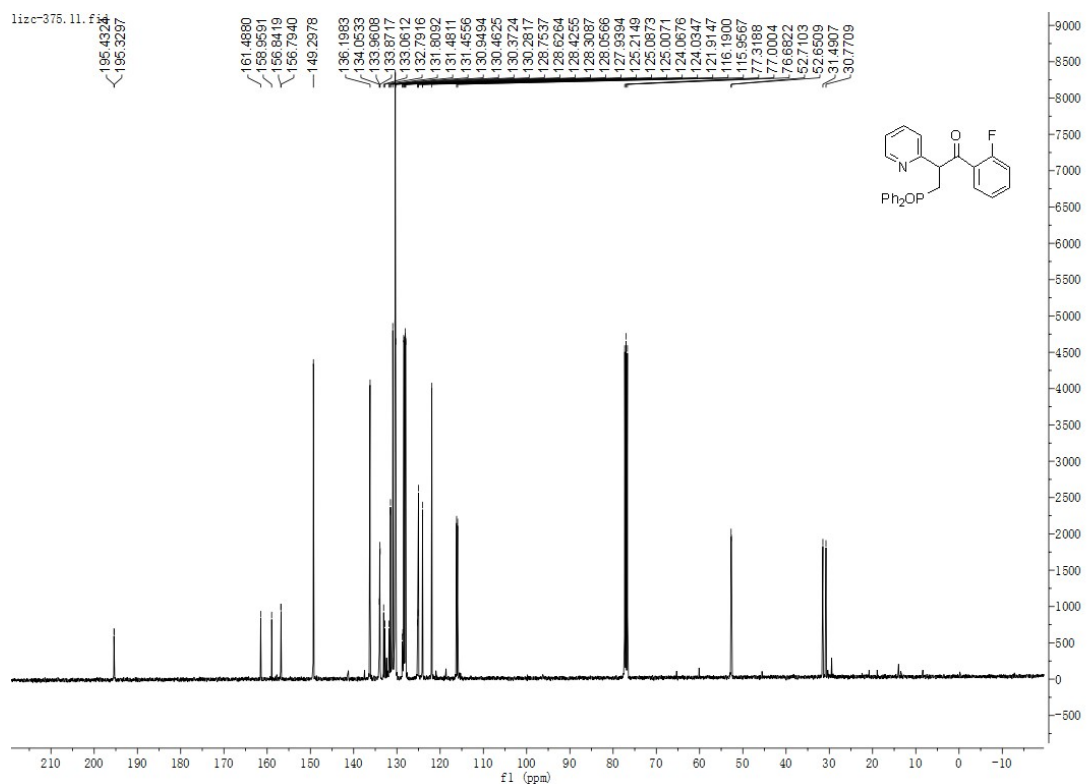


## 11. 3-(diphenylphosphoryl)-1-(2-fluorophenyl)-2-(pyridin-2-yl)propan-1-one (4k)

### <sup>1</sup>H NMR

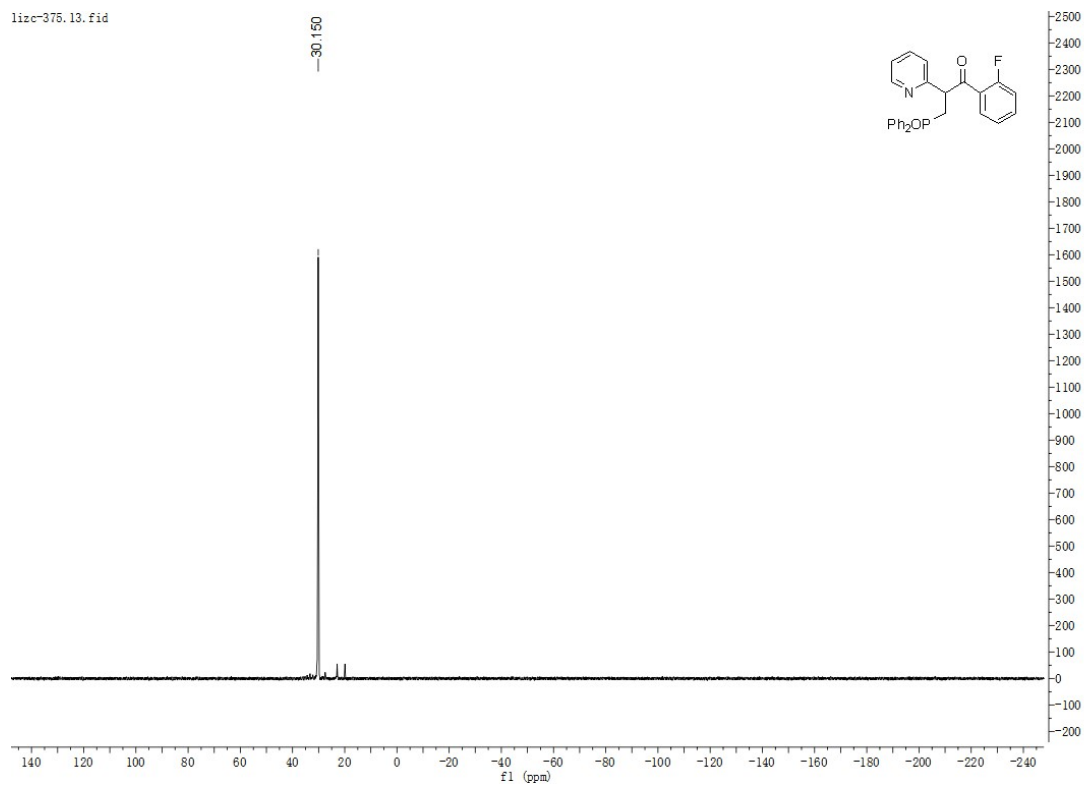


### <sup>13</sup>C NMR



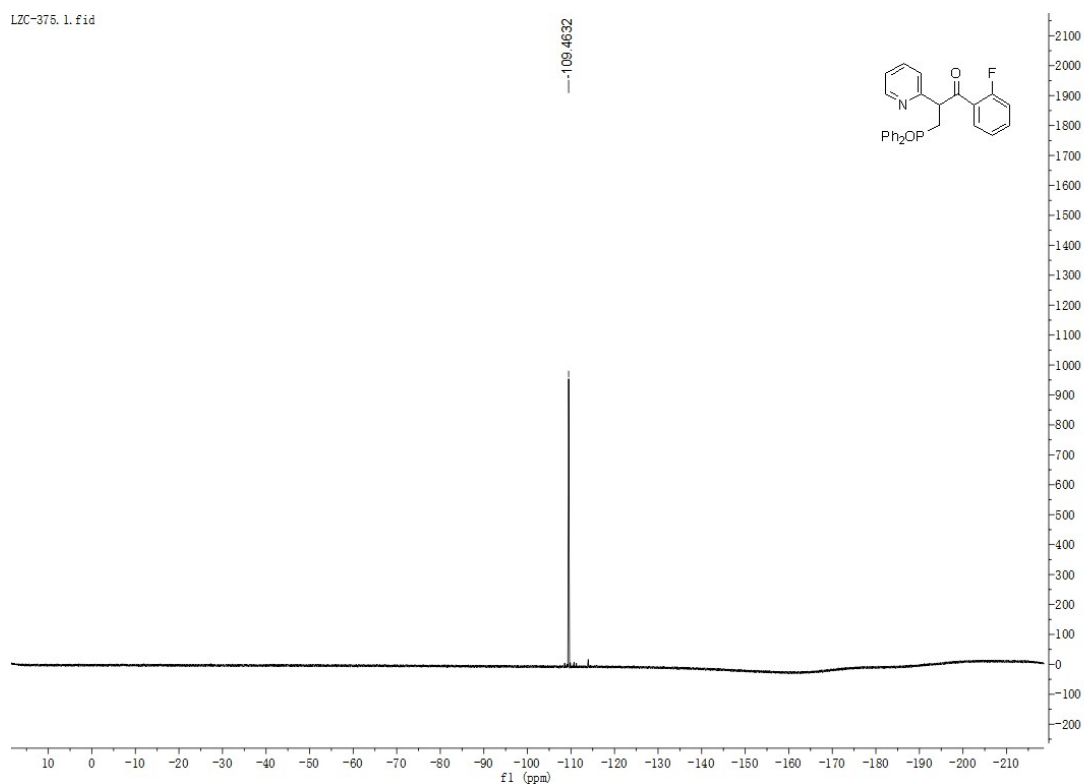
### <sup>31</sup>P NMR

lzc-375.13.fid



<sup>19</sup>F NMR

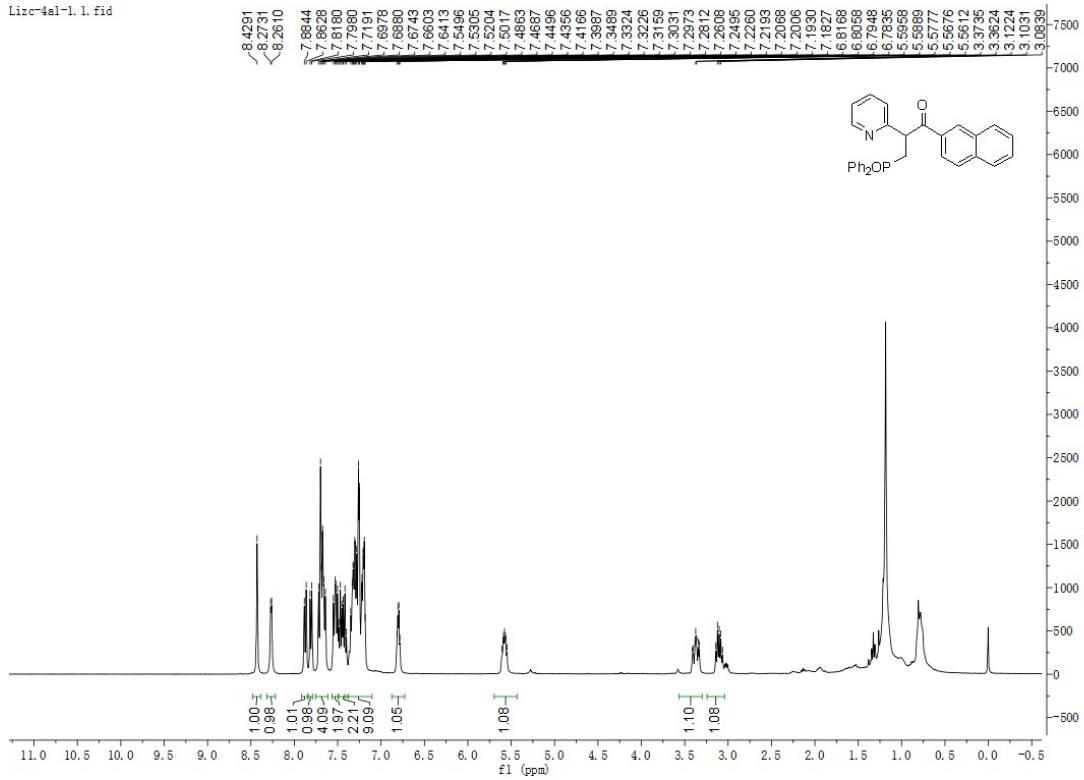
LZC-375.1.fid



**12. 3-(diphenylphosphoryl)-1-(naphthalen-2-yl)-2-(pyridin-2-yl)propan-1-one (4a)**

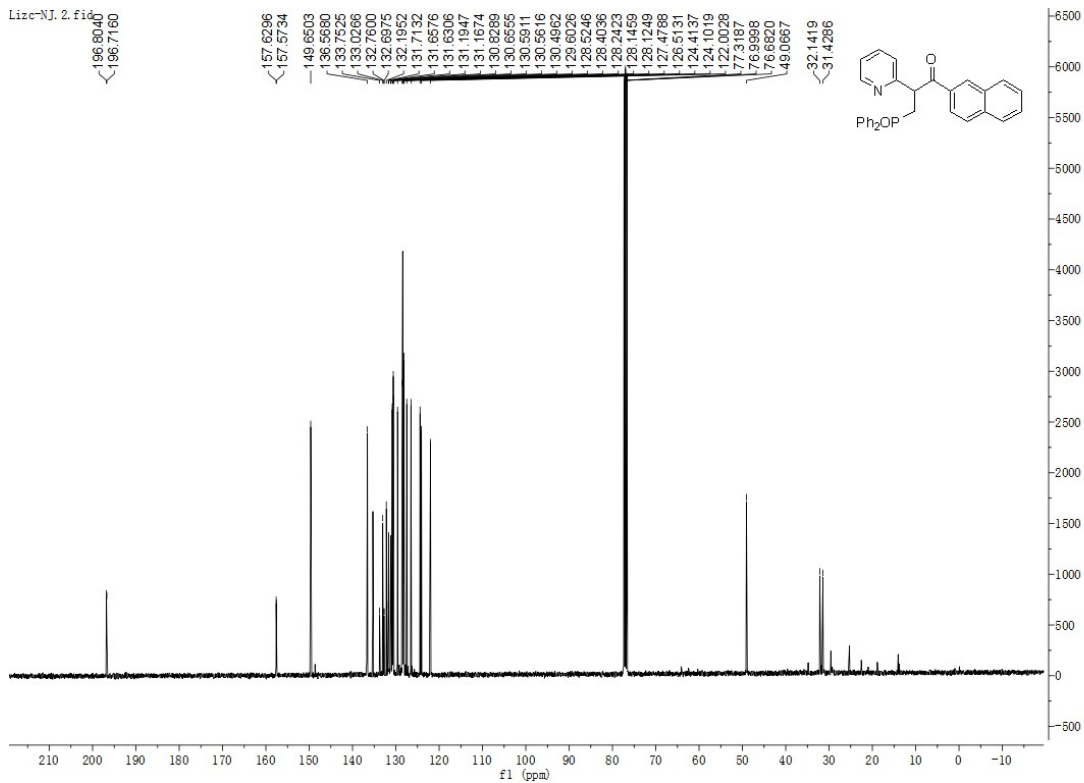
<sup>1</sup>H NMR

Lizc-4al-1.1.fid



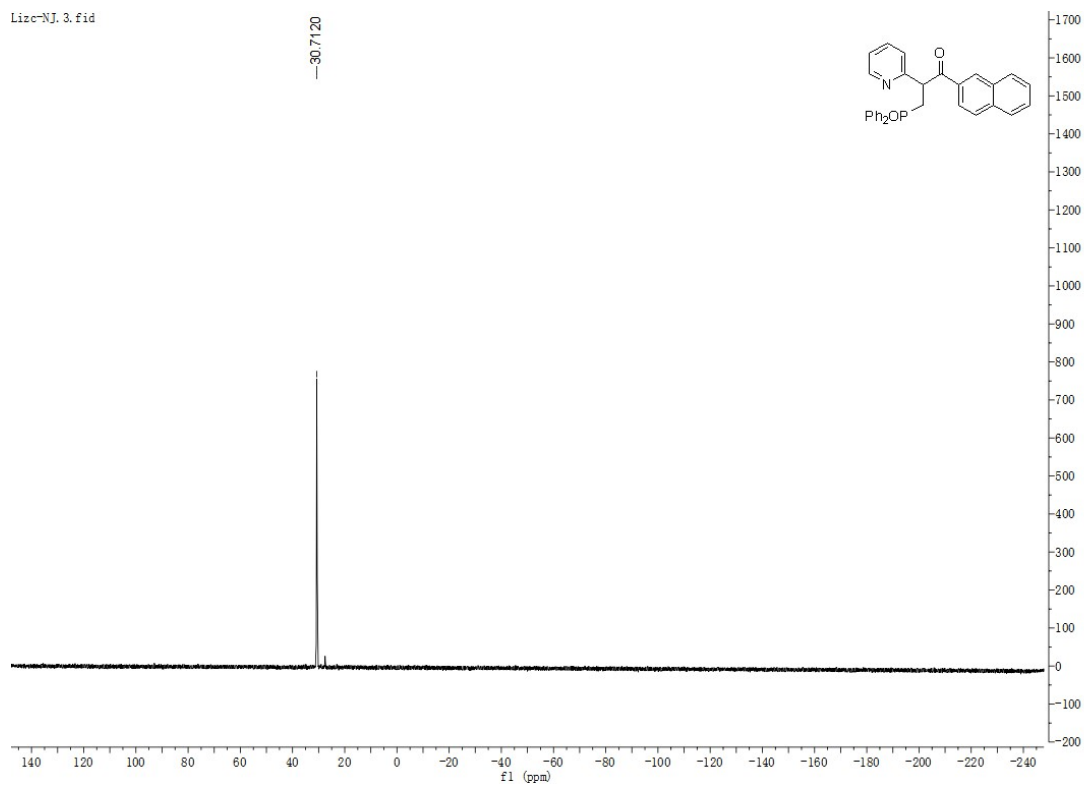
### <sup>13</sup>C NMR

Lizc-NJ.2.fid



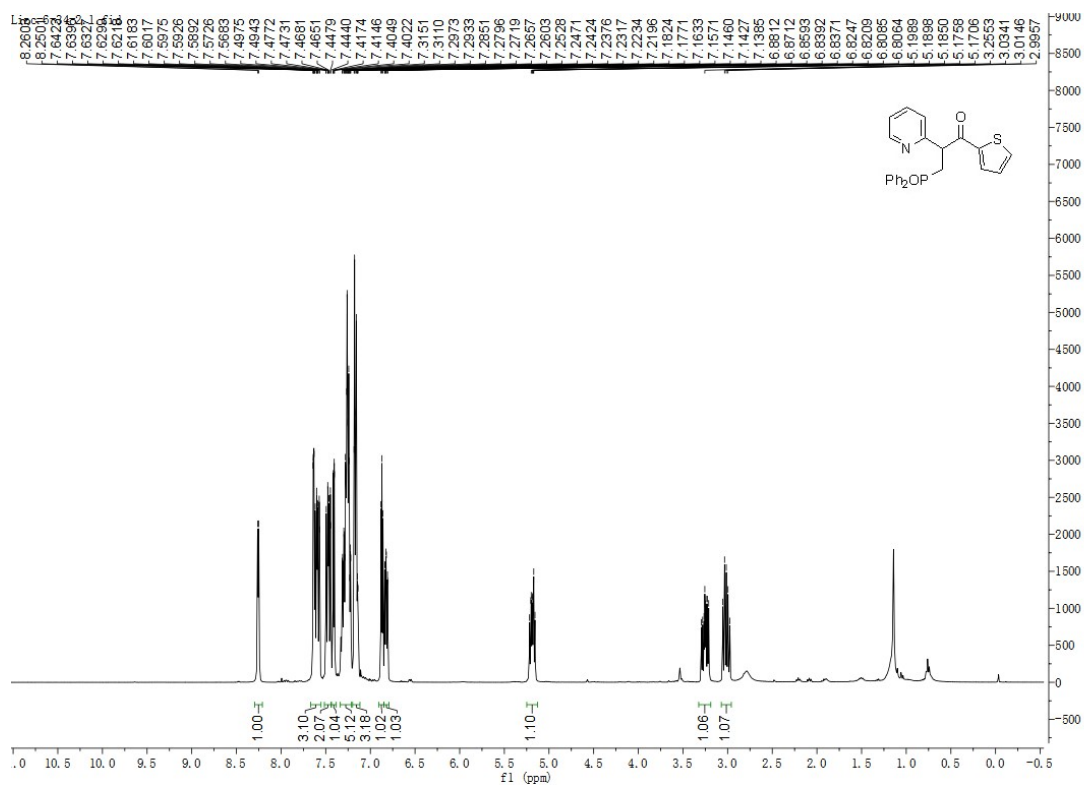
### <sup>31</sup>P NMR

Lize-NJ. 3. fid

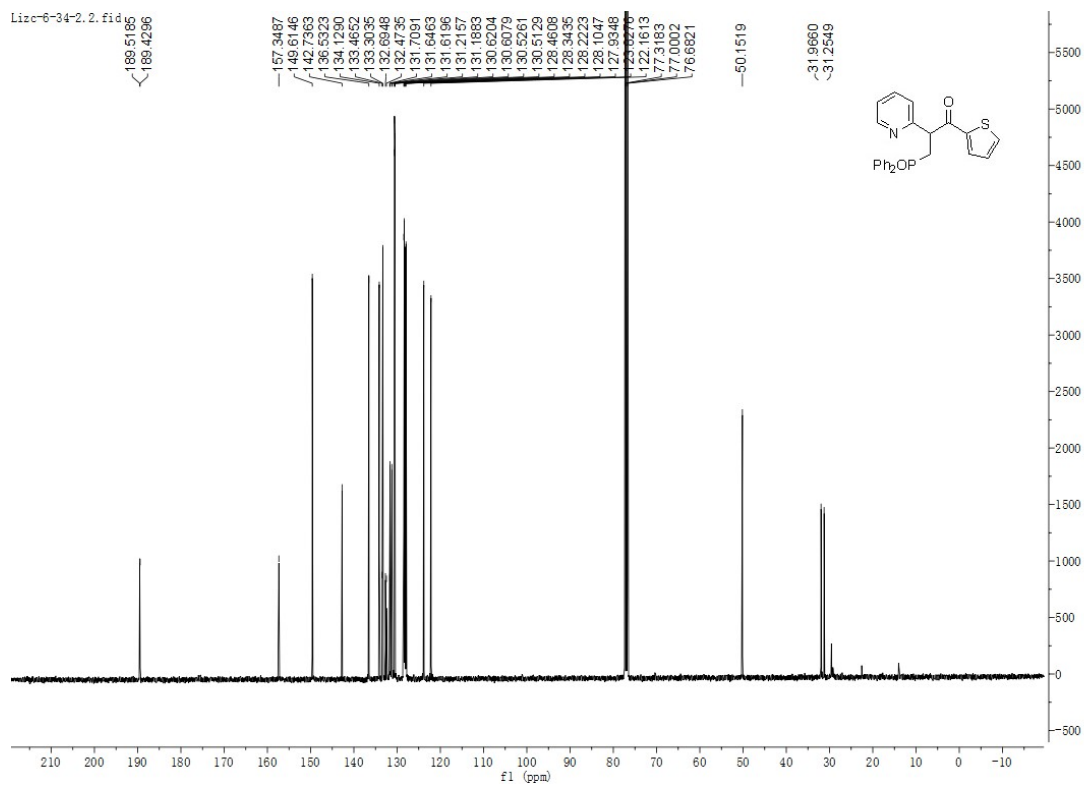


### 13. 3-(diphenylphosphoryl)-2-(pyridin-2-yl)-1-(thiophen-2-yl)propan-1-one (4am)

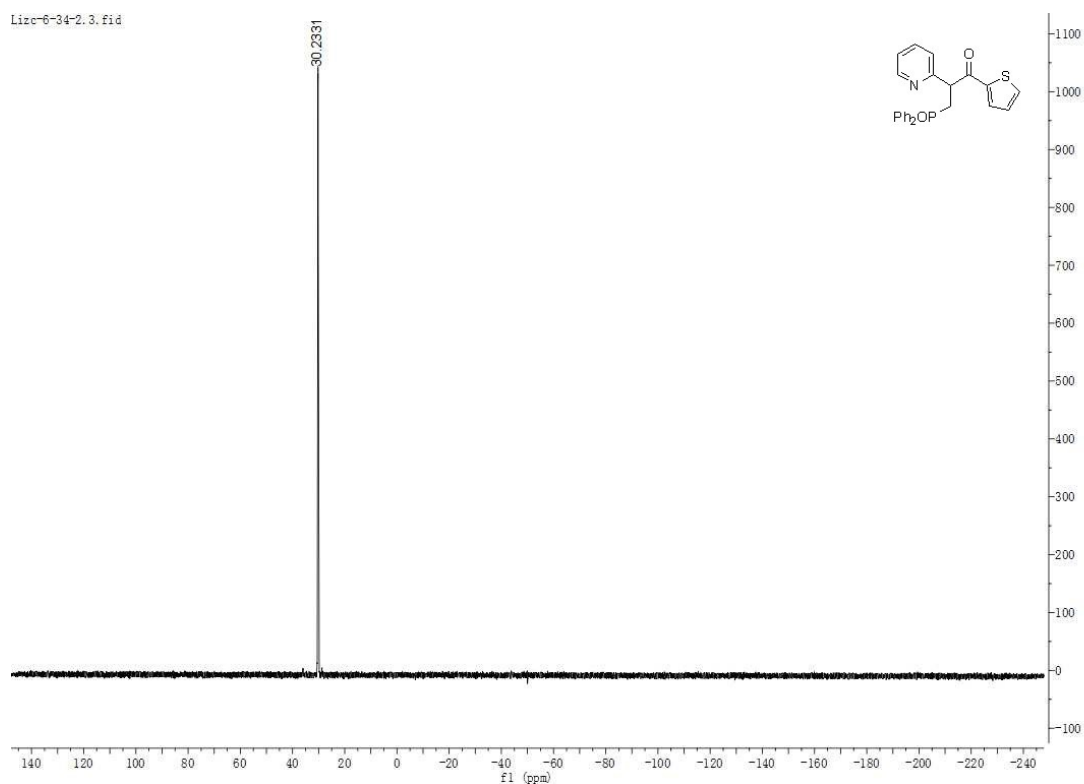
<sup>1</sup>H NMR



<sup>13</sup>C NMR

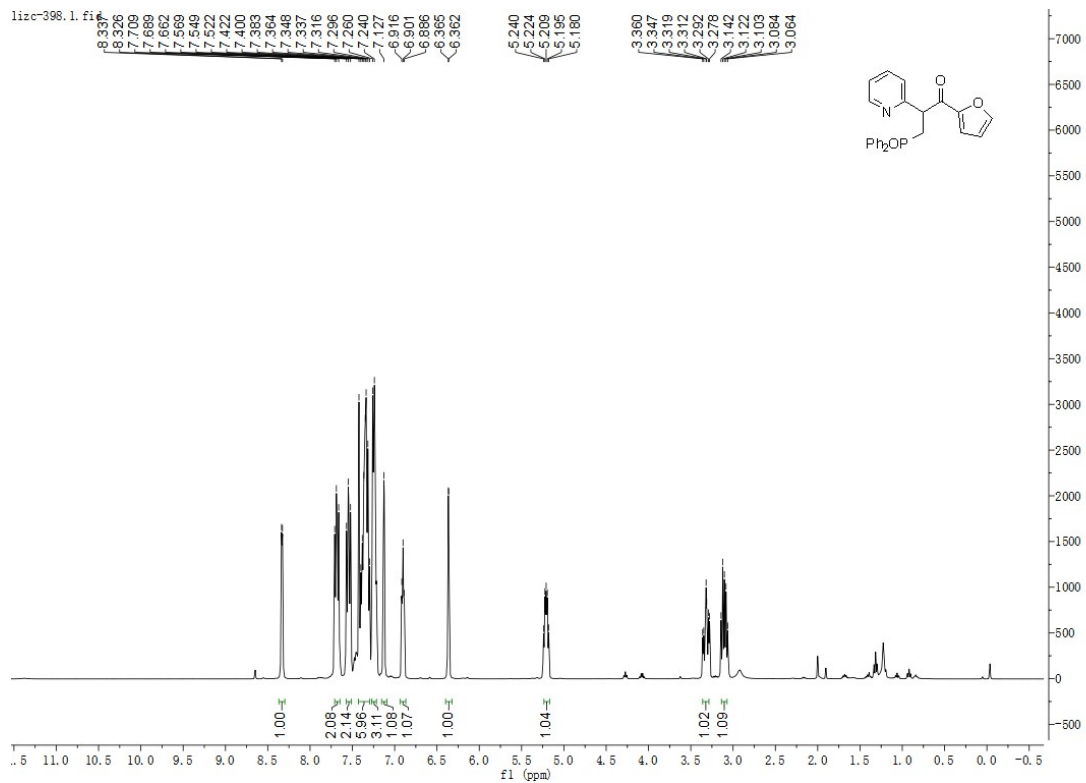


### <sup>31</sup>P NMR

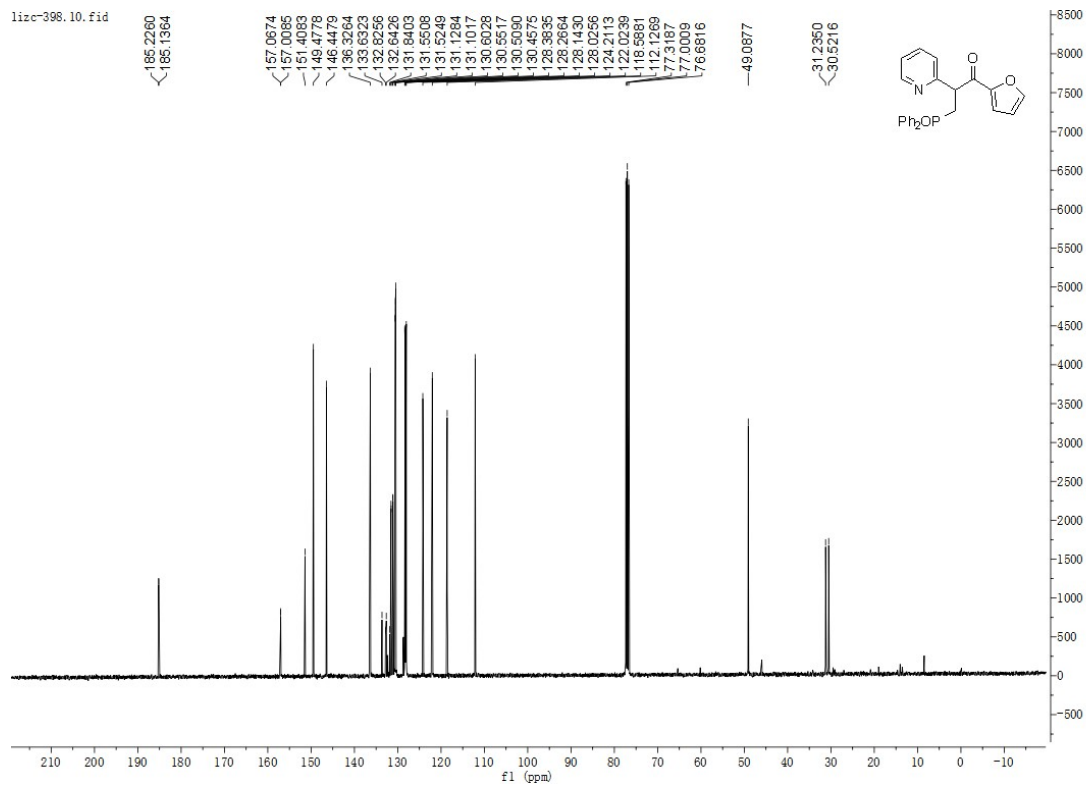


## 14. 3-(diphenylphosphoryl)-1-(furan-2-yl)-2-(pyridin-2-yl)propan-1-one (4an)

### <sup>1</sup>H NMR

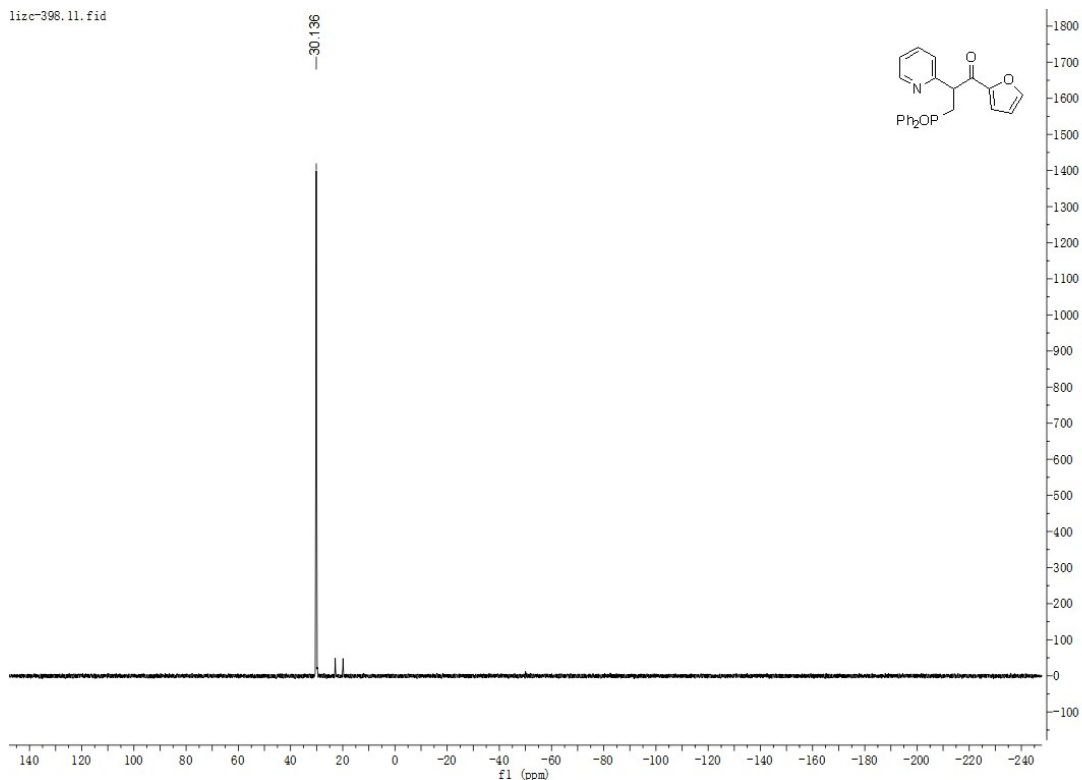


### <sup>13</sup>C NMR



### <sup>31</sup>P NMR

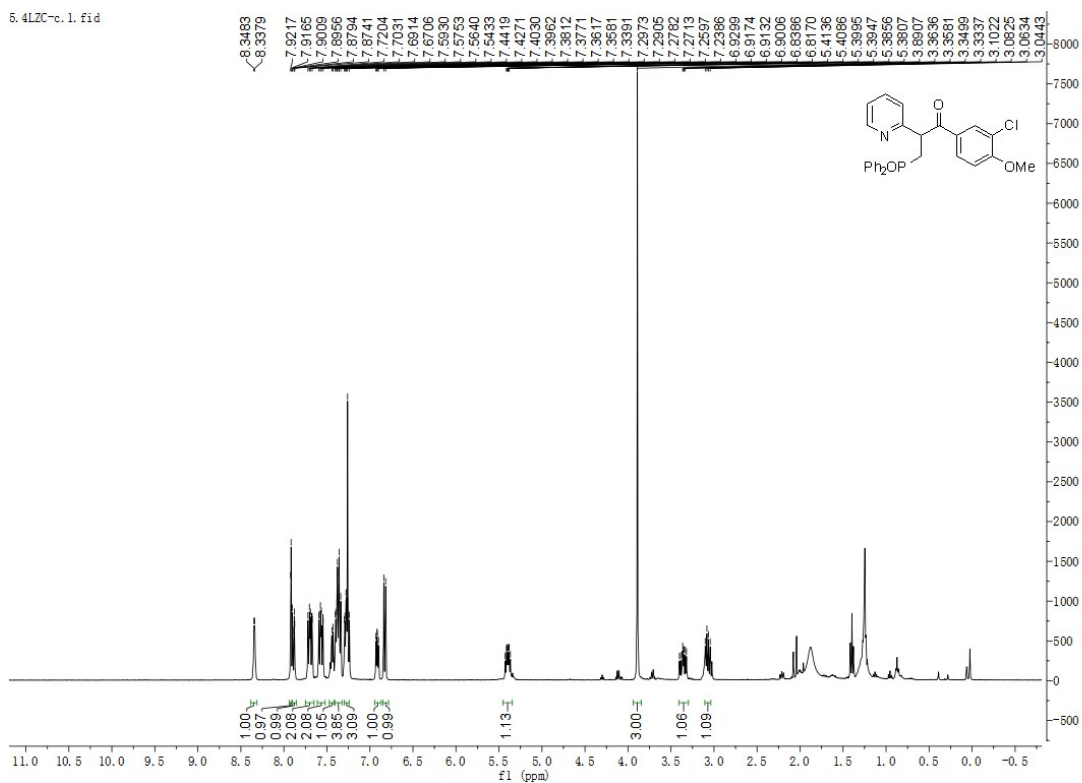
lizc-398.11.fid



### 15. 1-(3-chloro-4-methoxyphenyl)-3-(diphenylphosphoryl)-2-(pyridin-2-yl)propan-1-one (4ao)

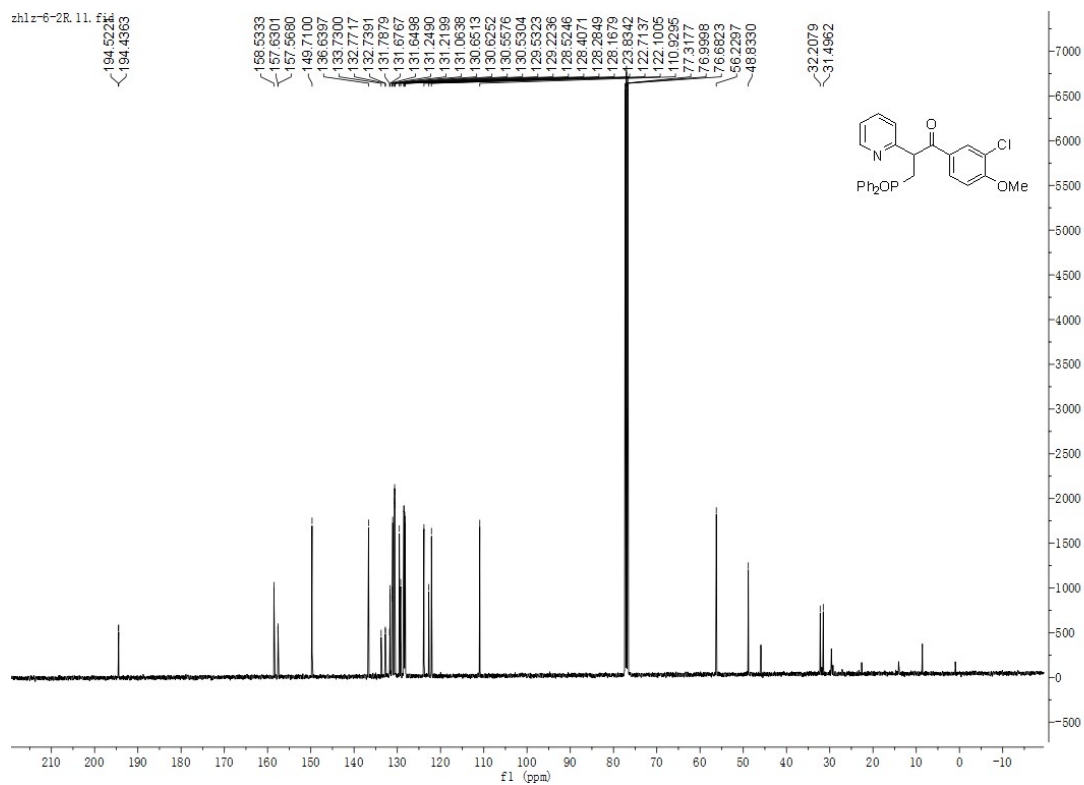
<sup>1</sup>H NMR

5.4LZC-c.1.fid

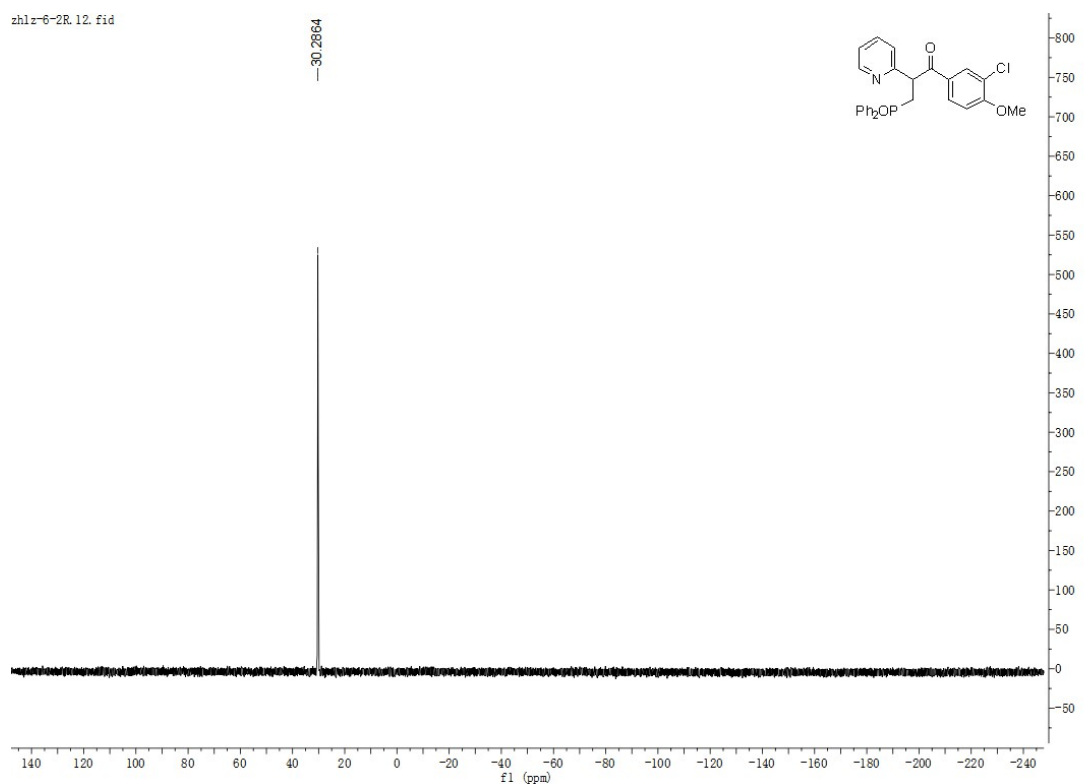


<sup>13</sup>C NMR



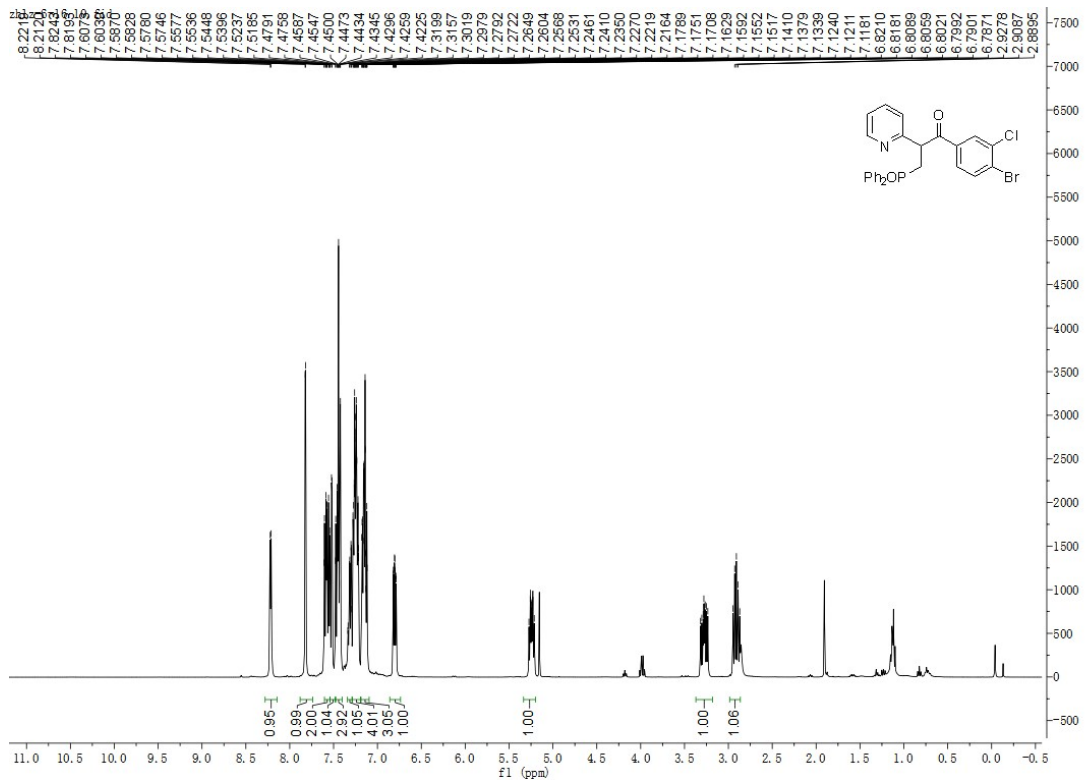


### <sup>31</sup>P NMR

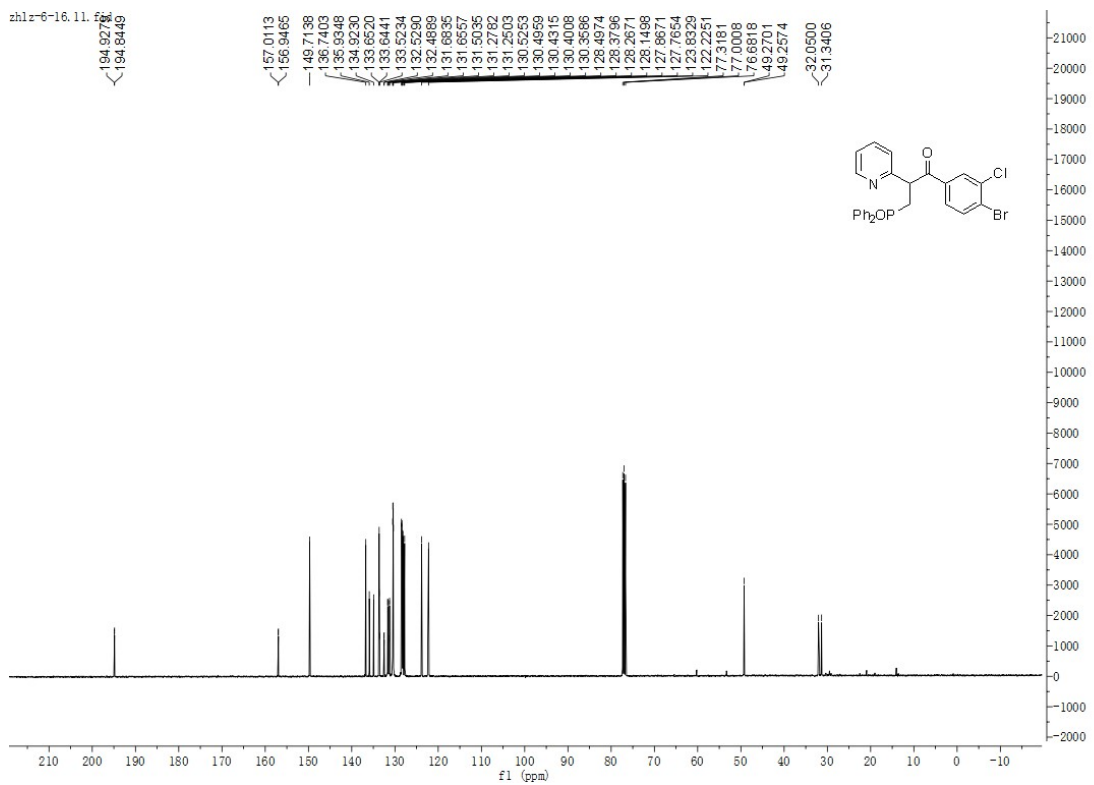


## 16. 1-(4-bromo-3-chlorophenyl)-3-(diphenylphosphoryl)-2-(pyridin-2-yl)propan-1-one (4ap)

### <sup>1</sup>H NMR

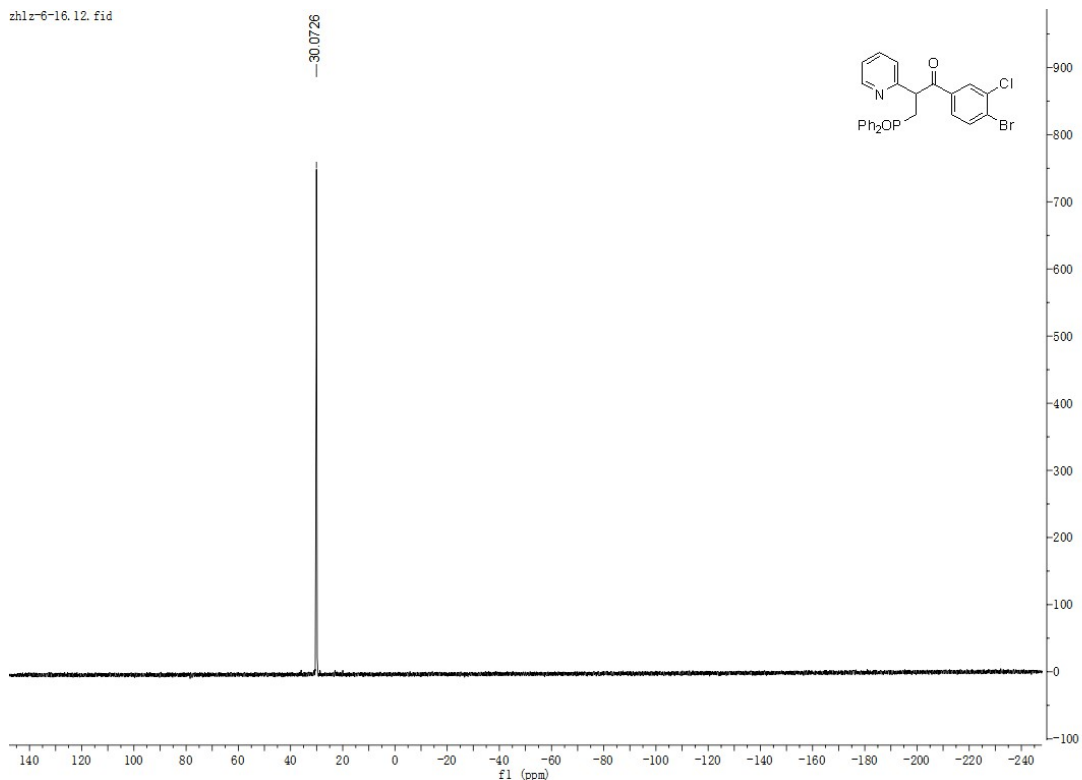


**<sup>13</sup>C NMR**



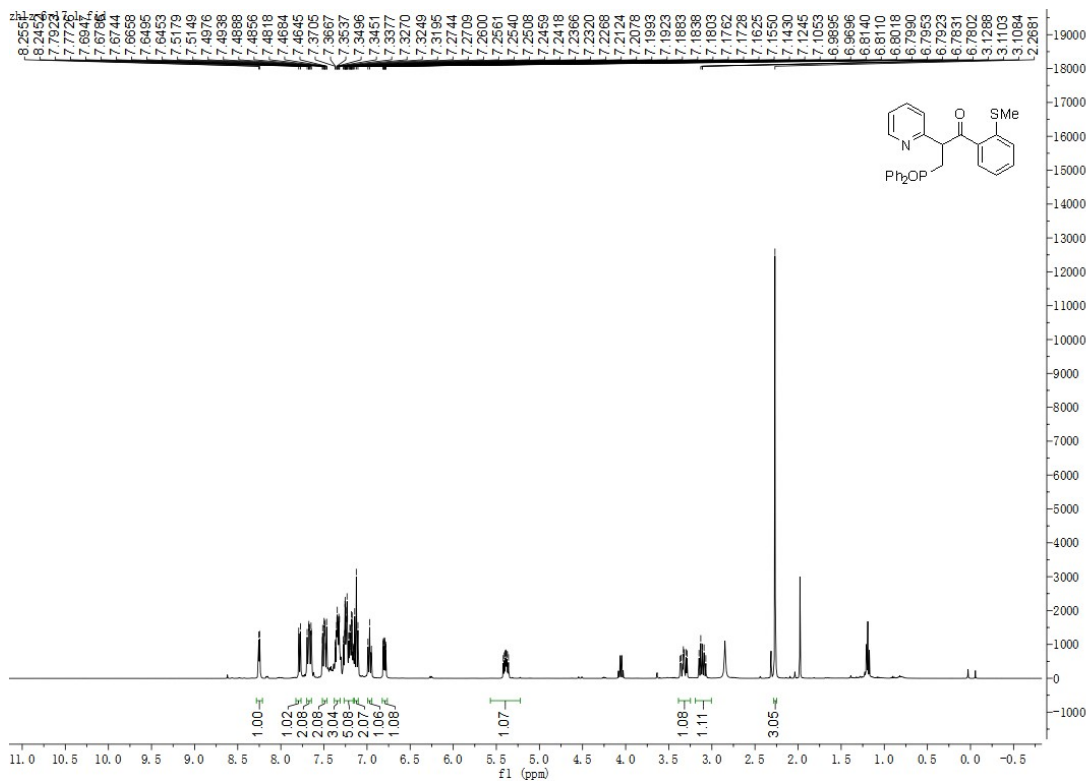
**<sup>31</sup>P NMR**

zhlz-6-16.12.fid

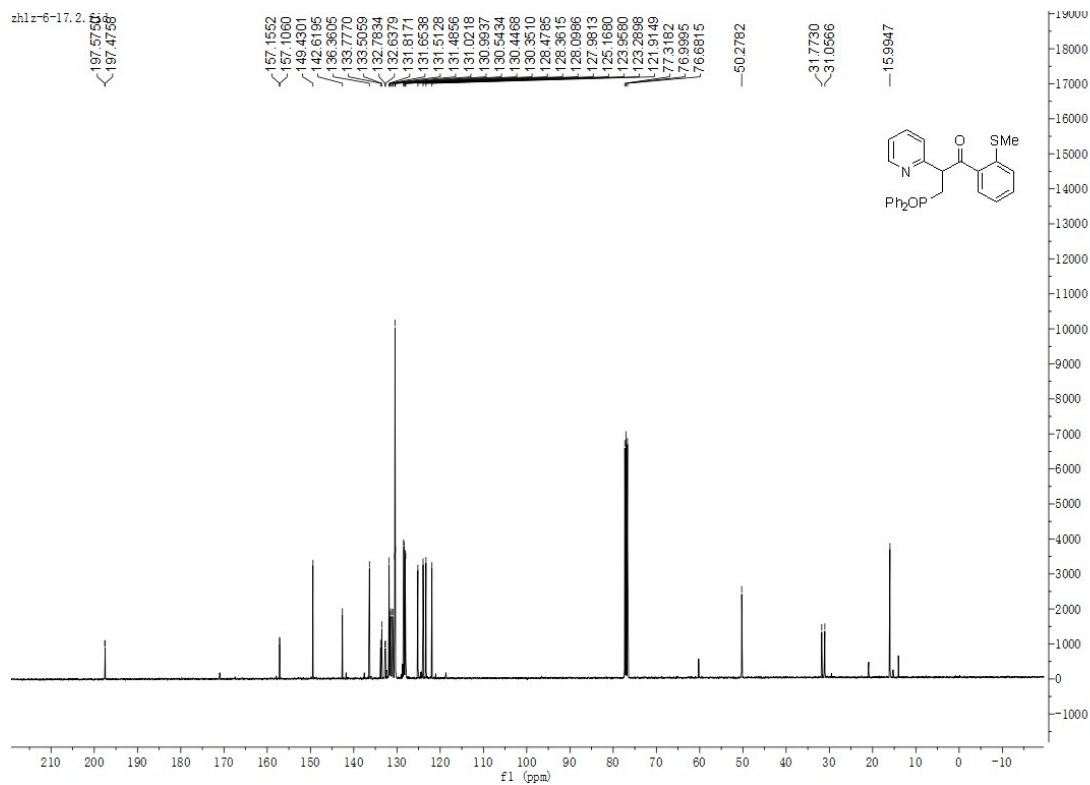


### 17. 3-(diphenylphosphoryl)-1-(2-(methylthio)phenyl)-2-(pyridin-2-yl)propan-1-one (4aq)

<sup>1</sup>H NMR

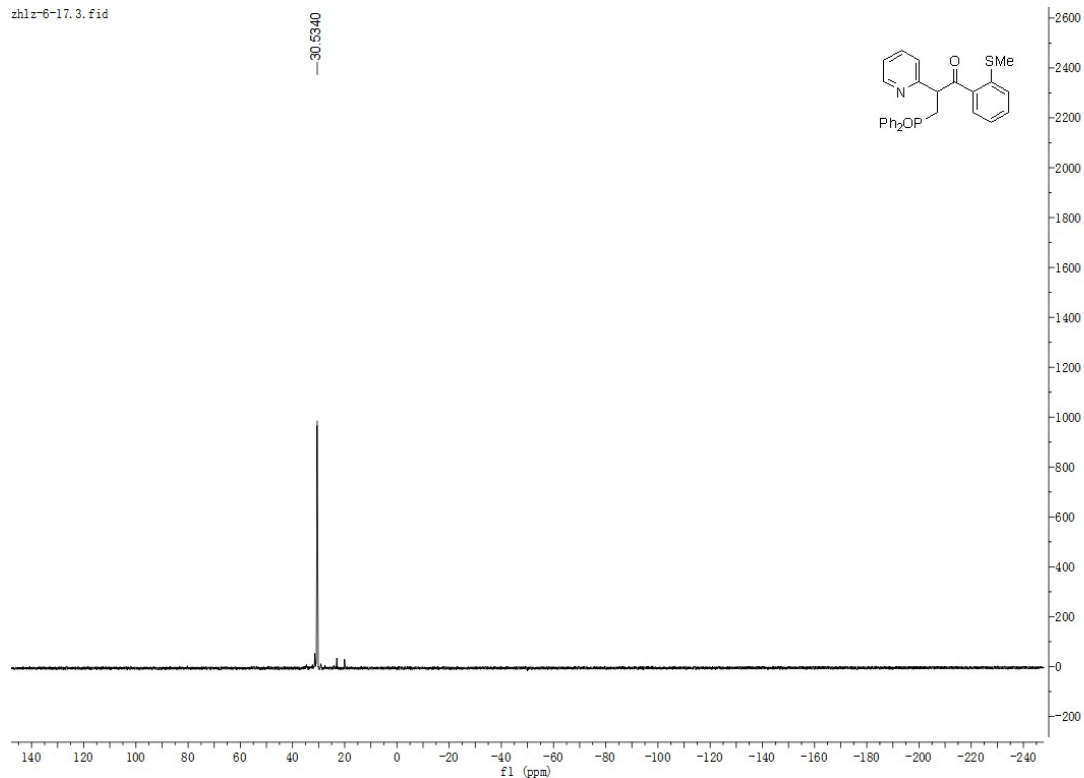


<sup>13</sup>C NMR



### <sup>31</sup>P NMR

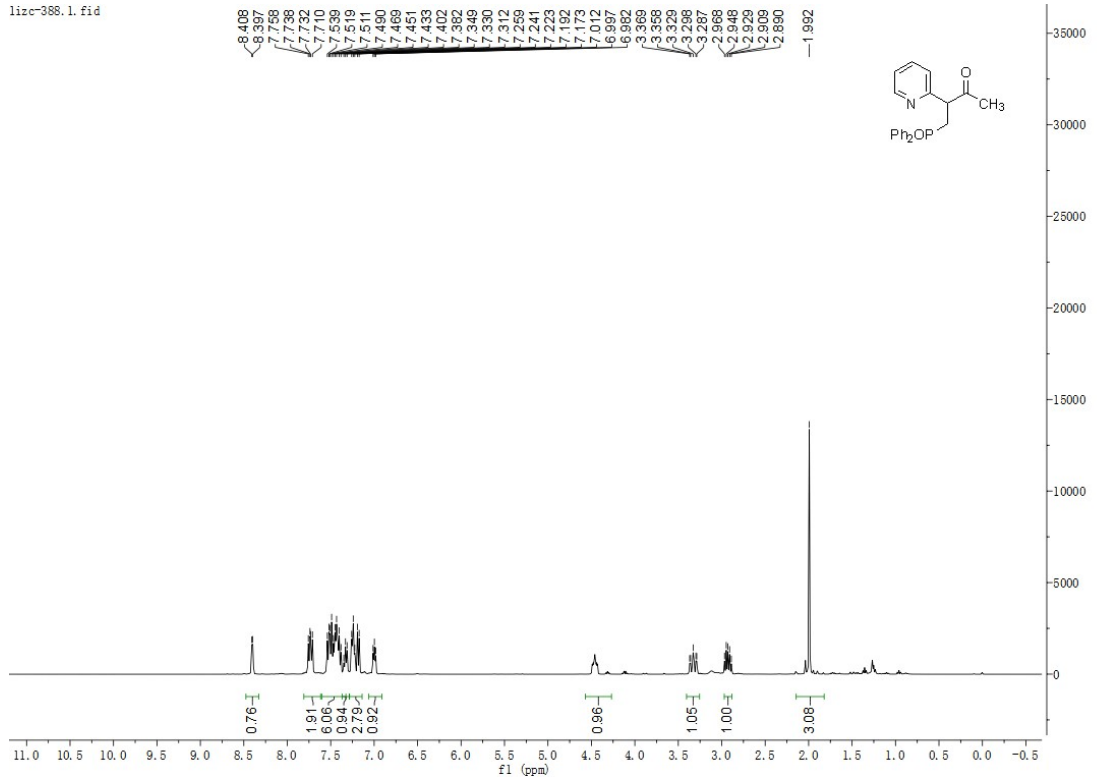
zhlz-6-17.3.fid



## 18. 4-(diphenylphosphoryl)-3-(pyridin-2-yl)butan-2-one (4ar)

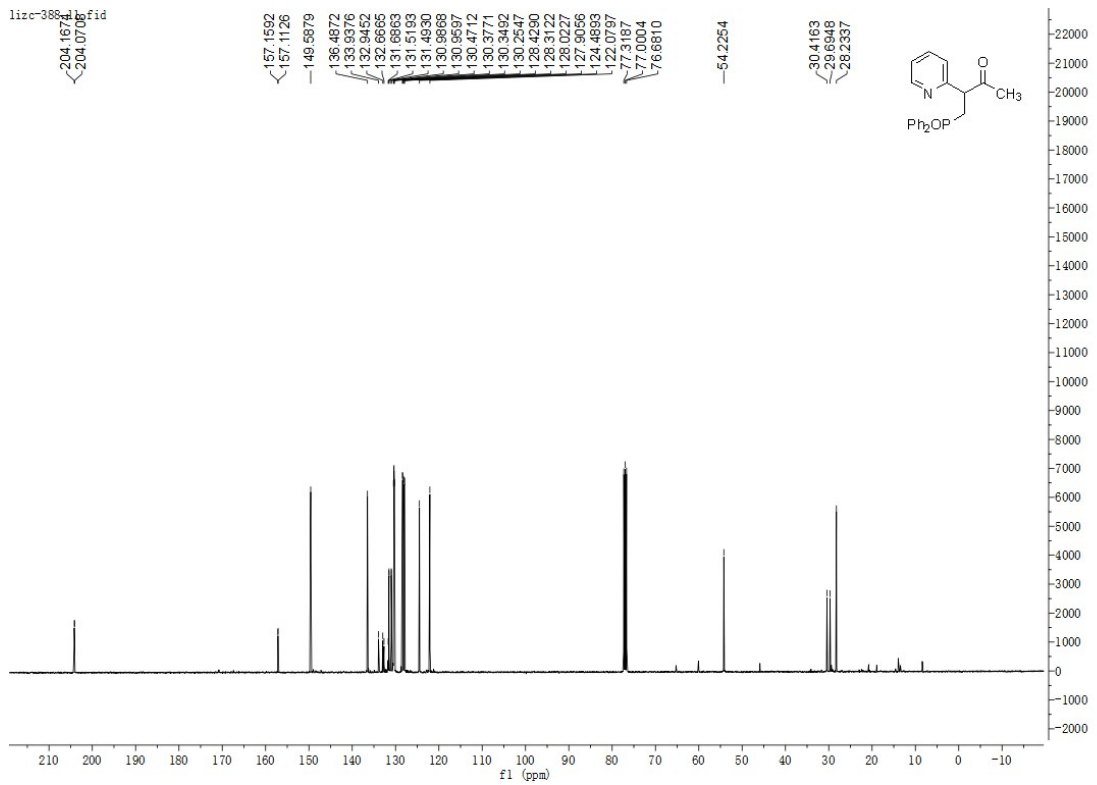
<sup>1</sup>H NMR

lizc-388.1.fid



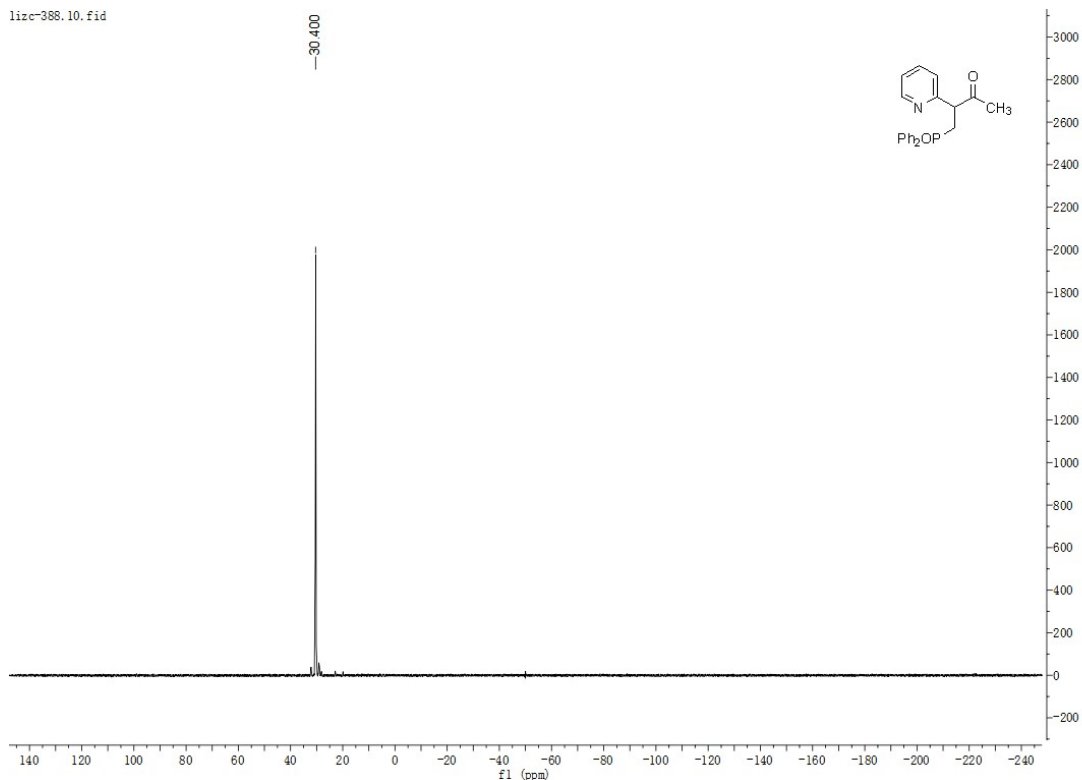
### <sup>13</sup>C NMR

lizc-388.1.fid



### <sup>31</sup>P NMR

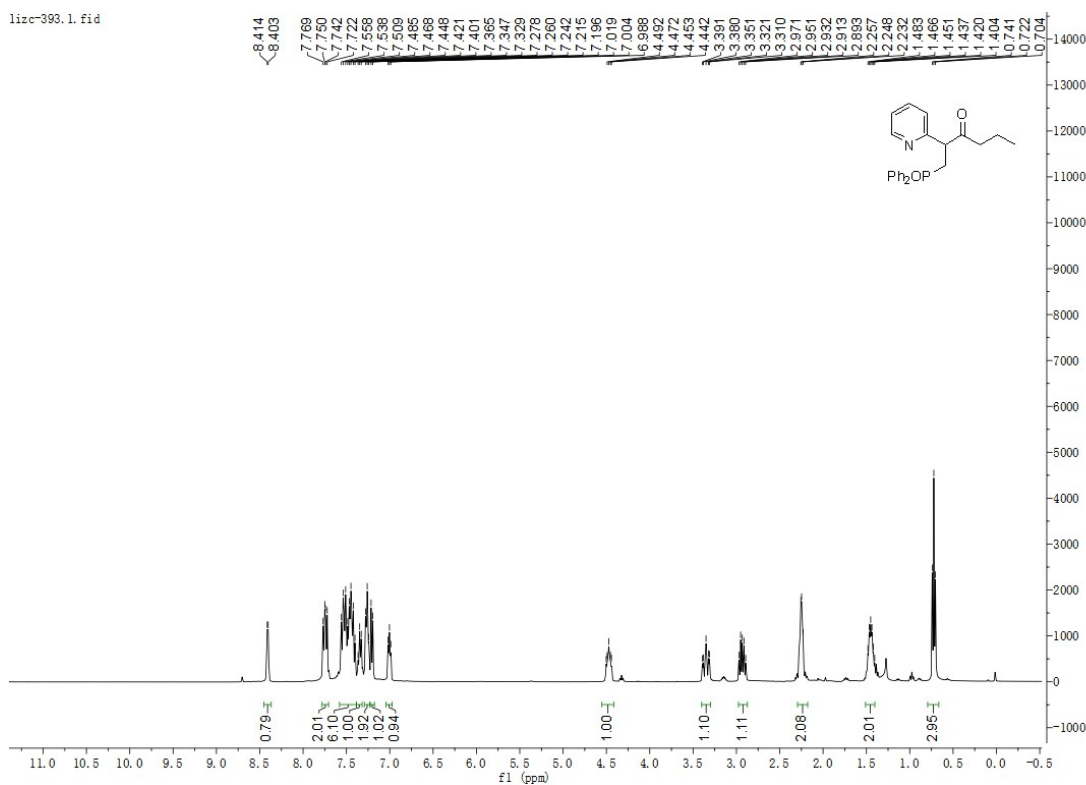
lizc-388.10.fid



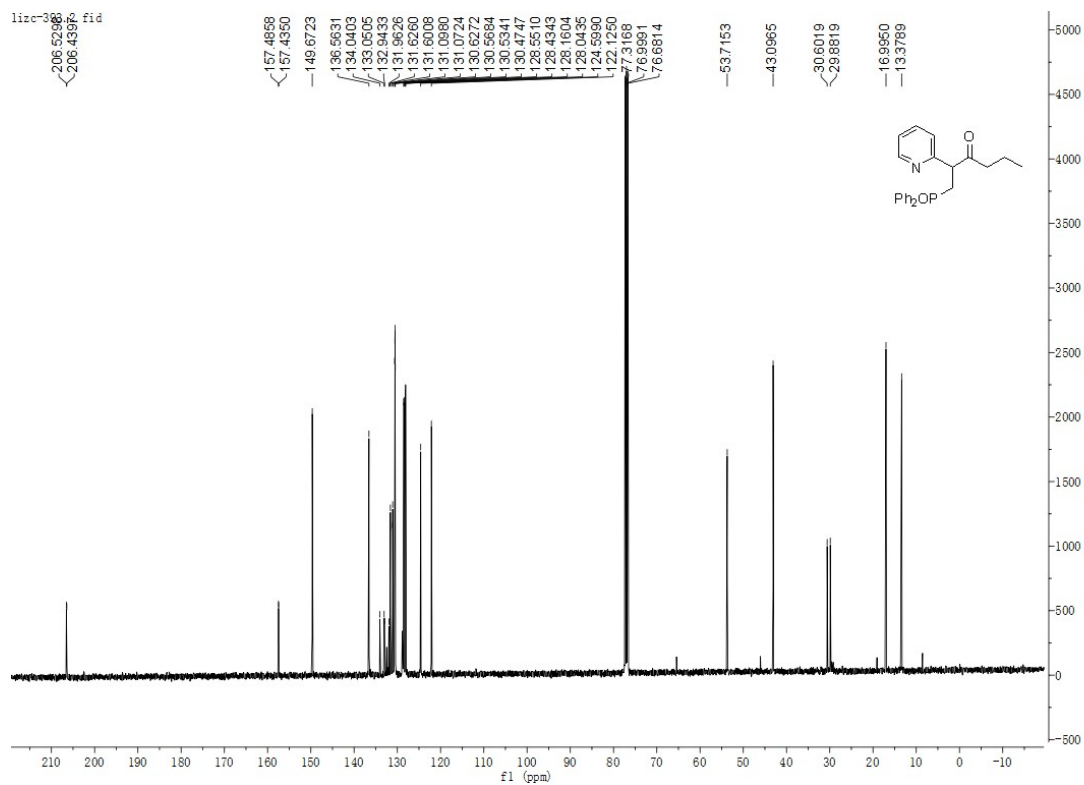
### 19. 1-(diphenylphosphoryl)-2-(pyridin-2-yl)hexan-3-one (4as)

<sup>1</sup>H NMR

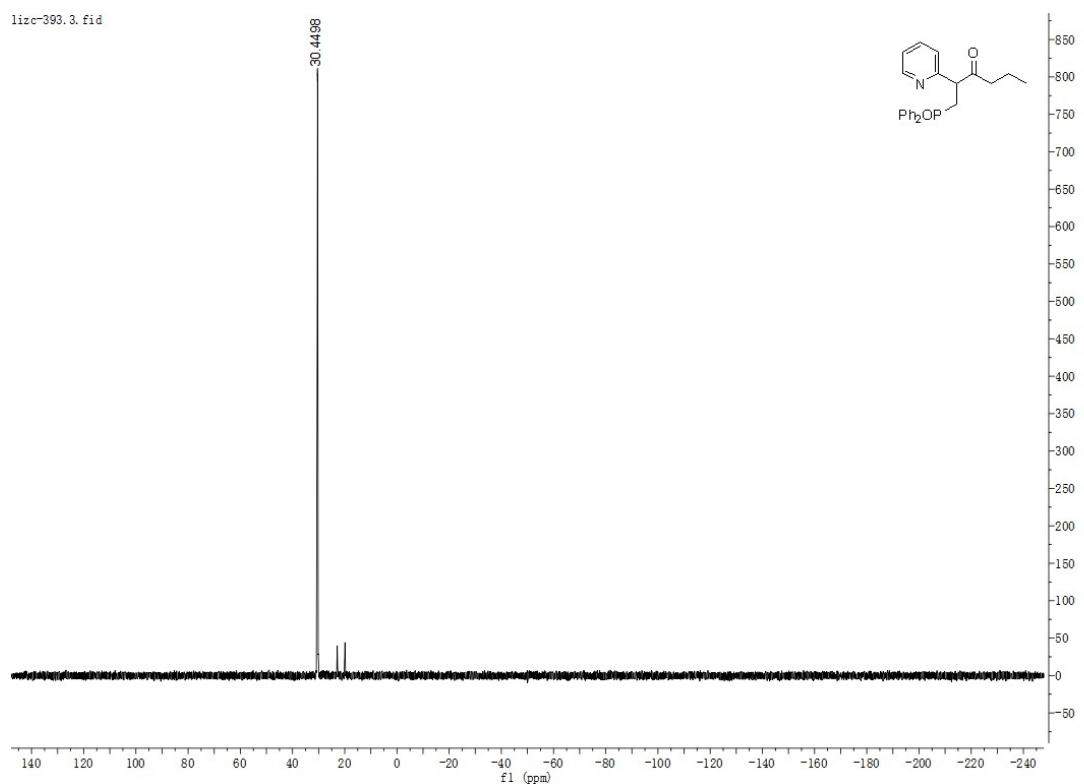
lizc-393.1.fid



<sup>13</sup>C NMR



### <sup>31</sup>P NMR



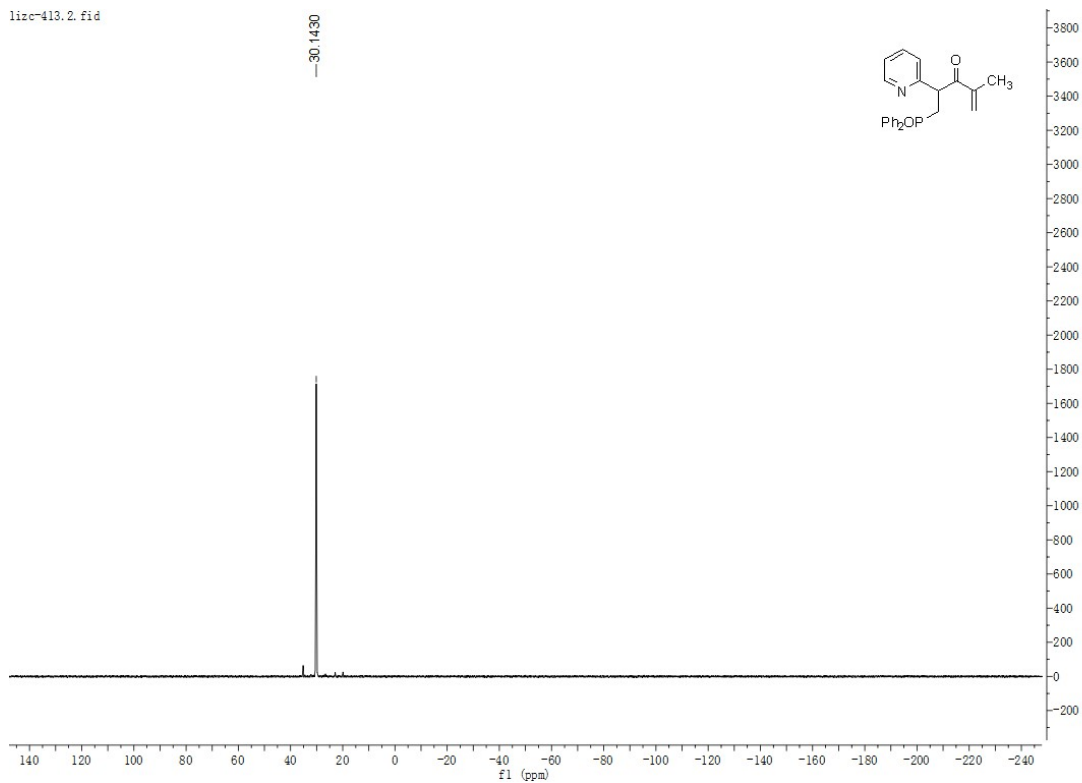
## 20. 5-(diphenylphosphoryl)-2-methyl-4-(pyridin-2-yl)pent-1-en-3-one (4at)

<sup>1</sup>H NMR



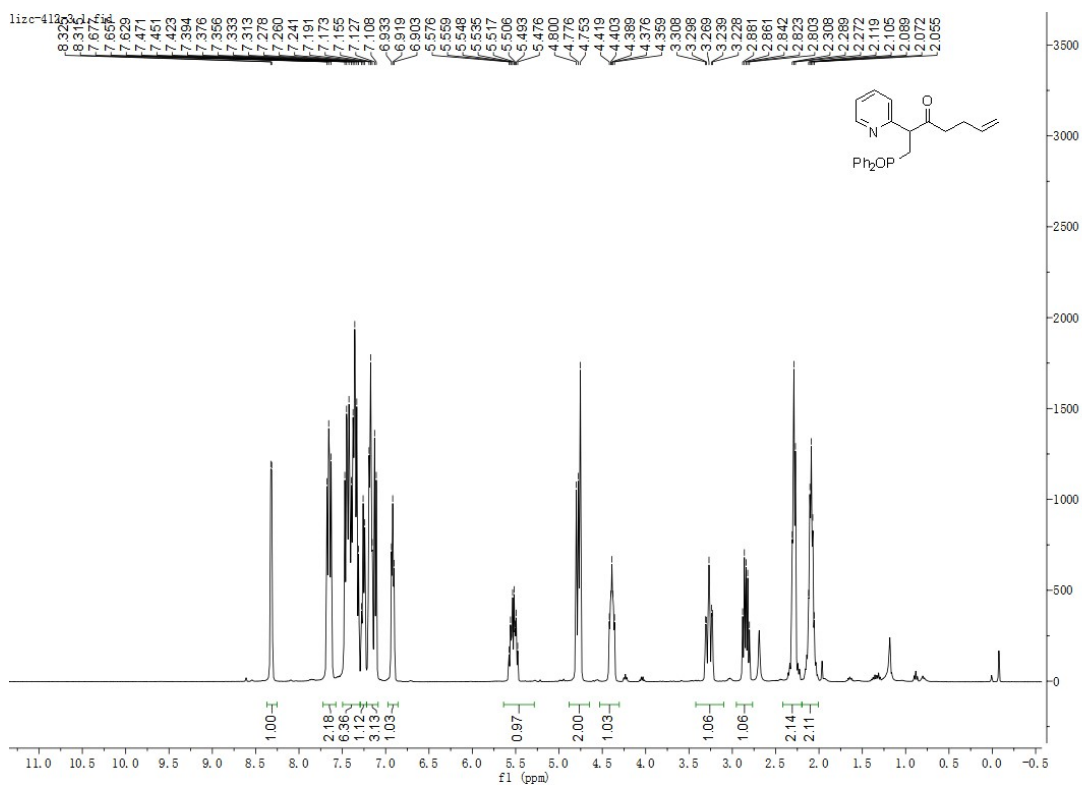


lizc-413.2.fid

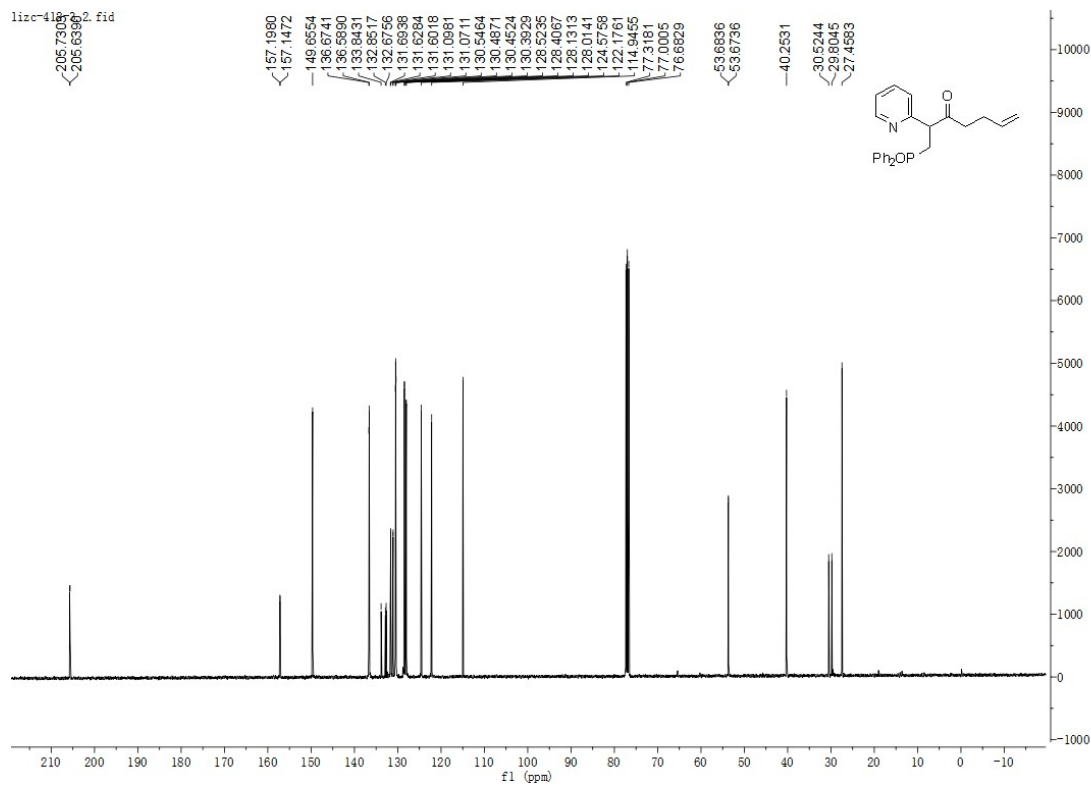


## 21. 1-(diphenylphosphoryl)-2-(pyridin-2-yl)hept-6-en-3-one (4au)

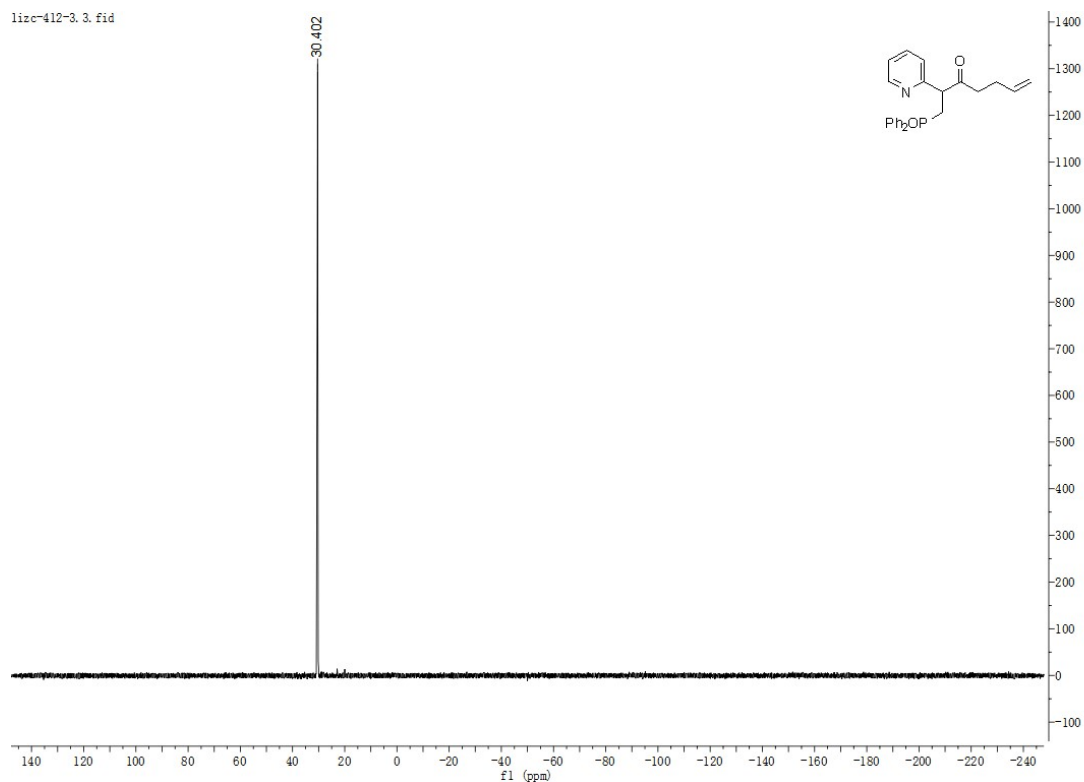
<sup>1</sup>H NMR



<sup>13</sup>C NMR

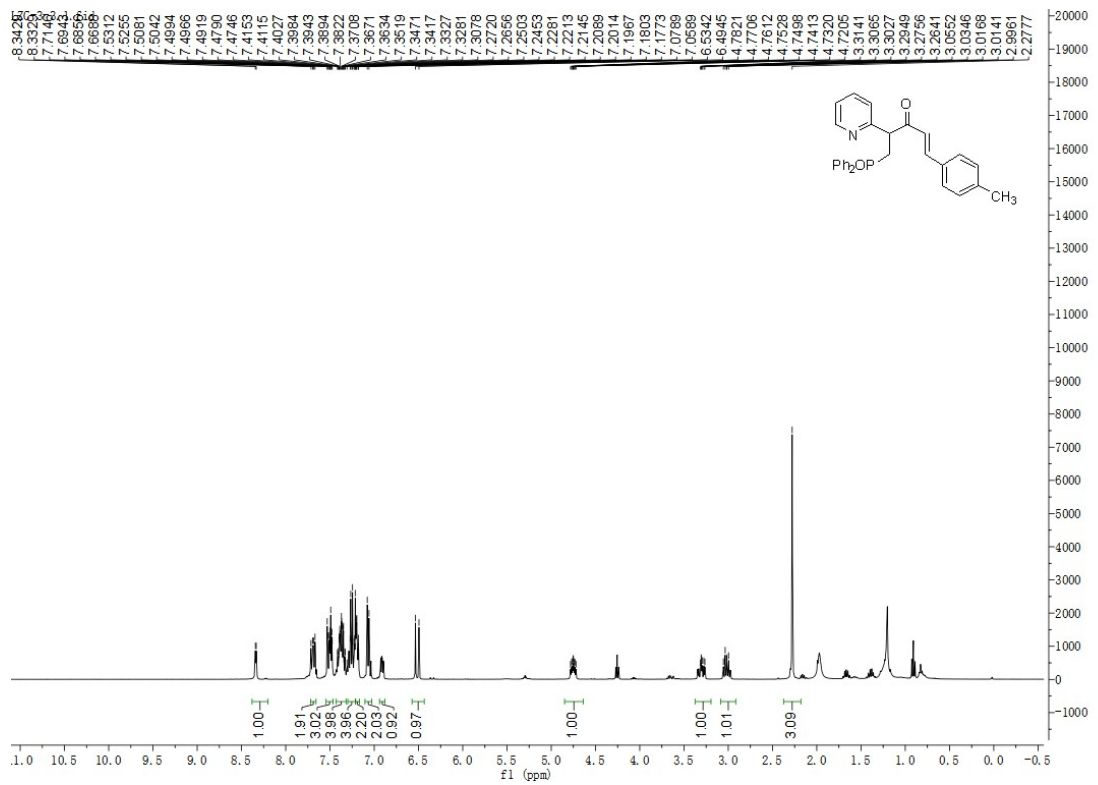


### <sup>31</sup>P NMR



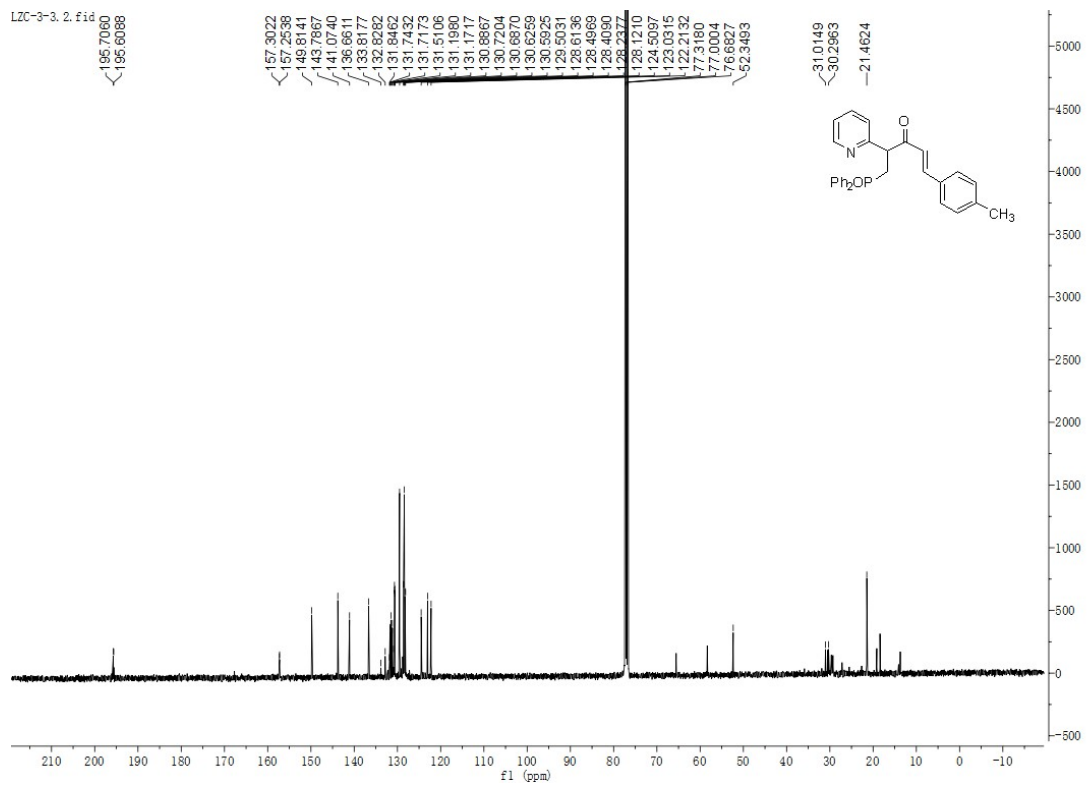
## 22. (E)-5-(diphenylphosphoryl)-4-(pyridin-2-yl)-1-(p-tolyl)pent-1-en-3-one (4av)

<sup>1</sup>H NMR



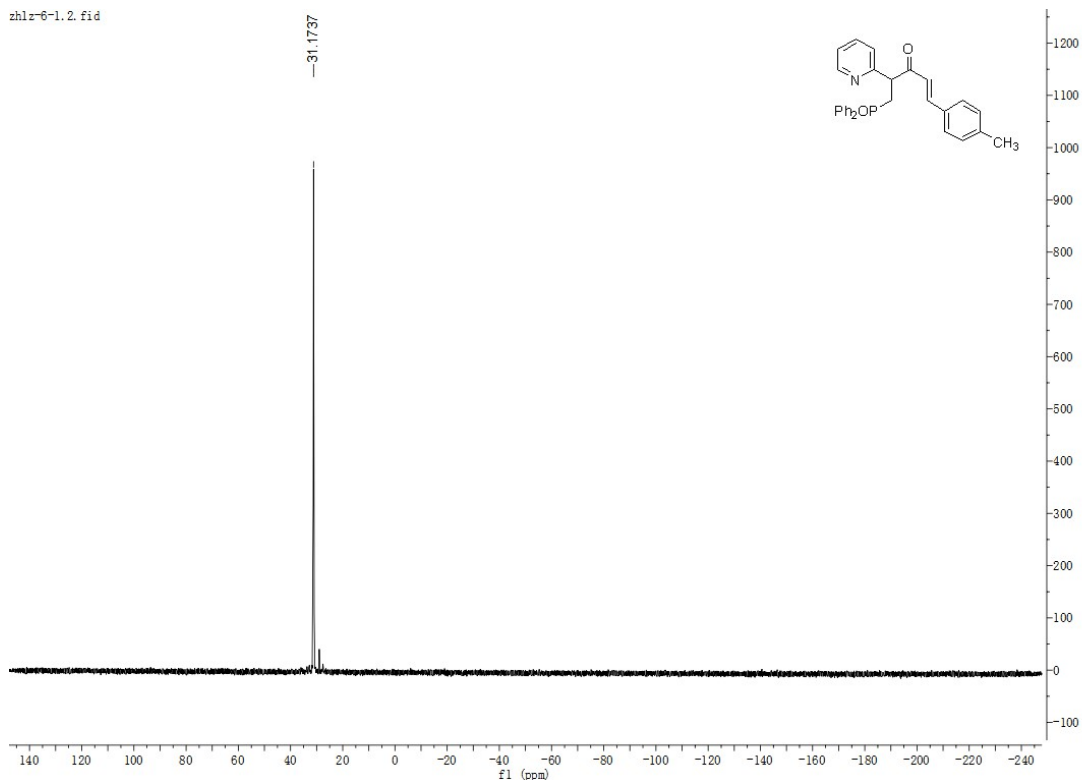
**<sup>13</sup>C NMR**

LZC-3-3. 2. f1.d



**<sup>31</sup>P NMR**

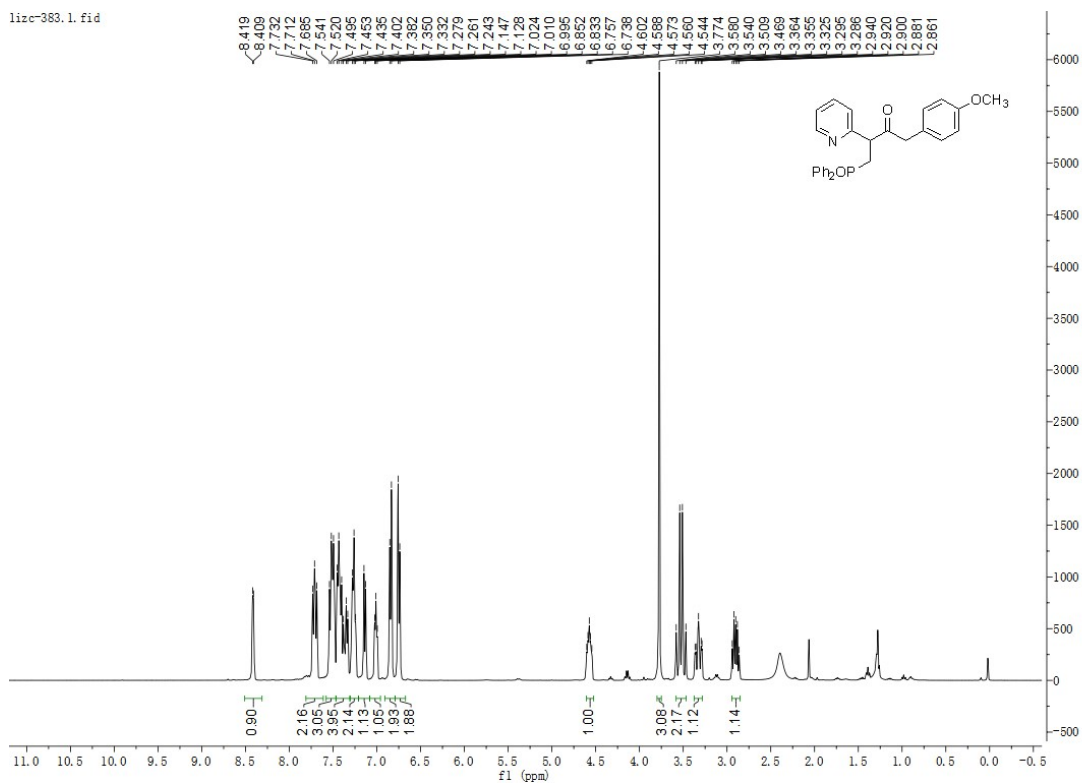
zh1z-6-1.2.fid



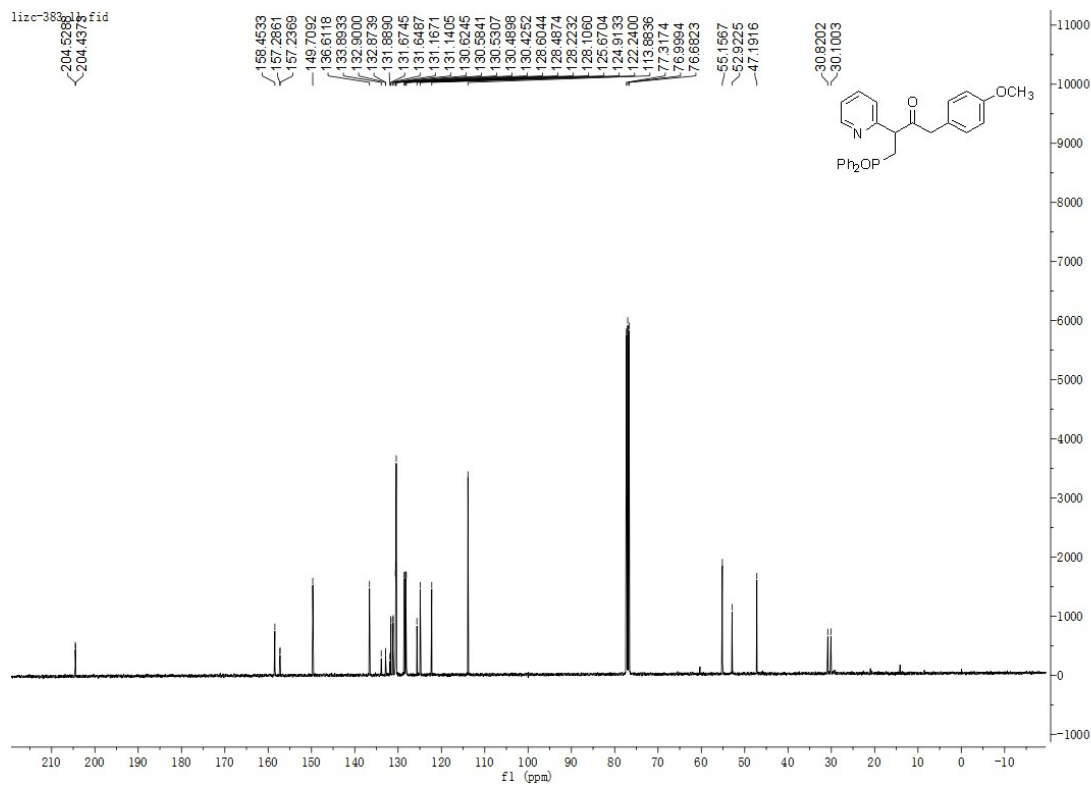
### 23. 4-(diphenylphosphoryl)-1-(4-methoxyphenyl)-3-(pyridin-2-yl)butan-2-one (4aw)

<sup>1</sup>H NMR

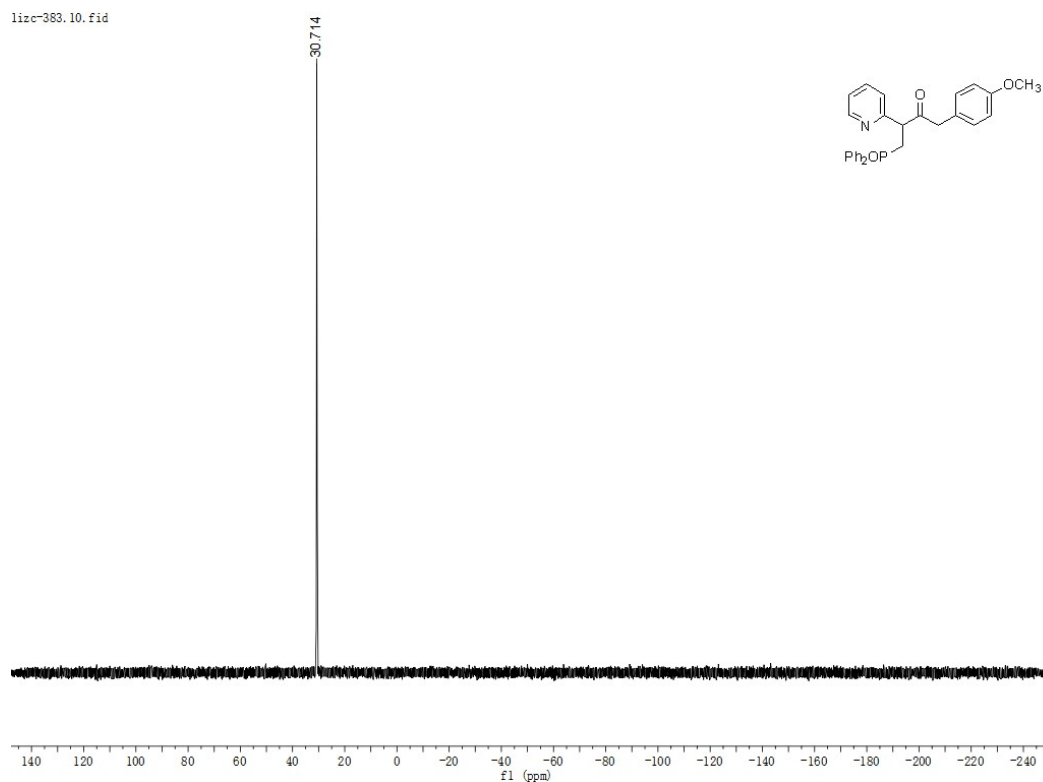
lize-383.1.fid



<sup>13</sup>C NMR



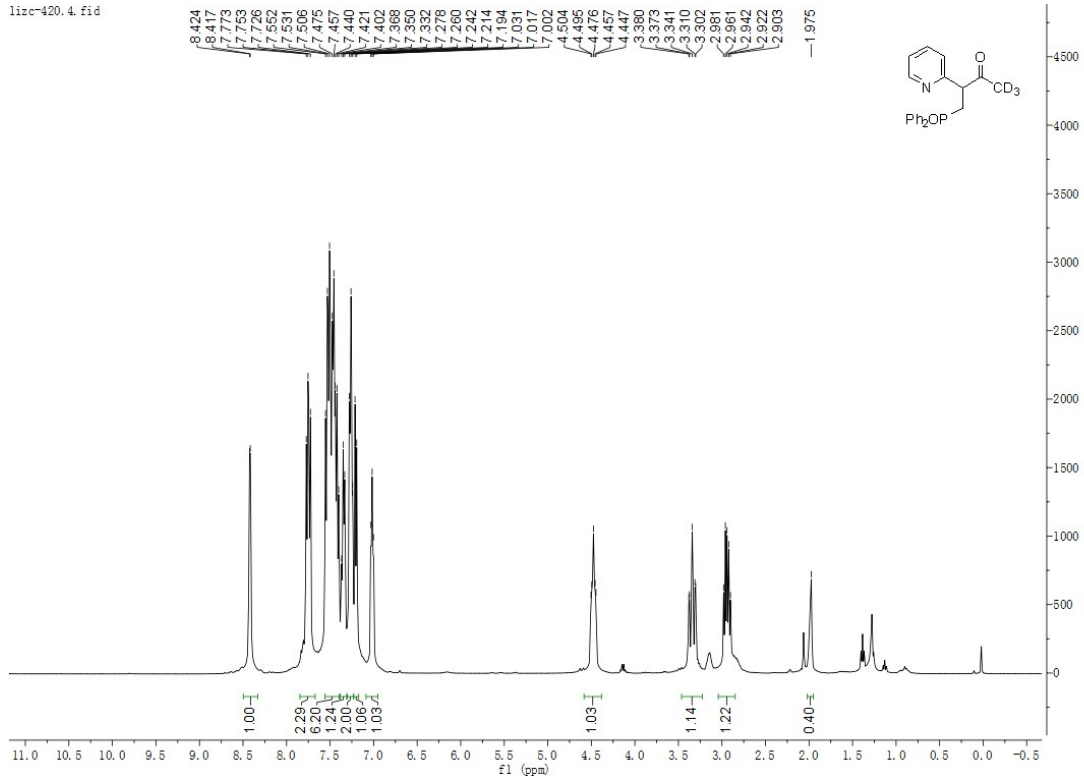
### <sup>31</sup>P NMR



## 24. 4-(diphenylphosphoryl)-3-(pyridin-2-yl)butan-2-one-1,1,1-d<sub>3</sub> (4ax)

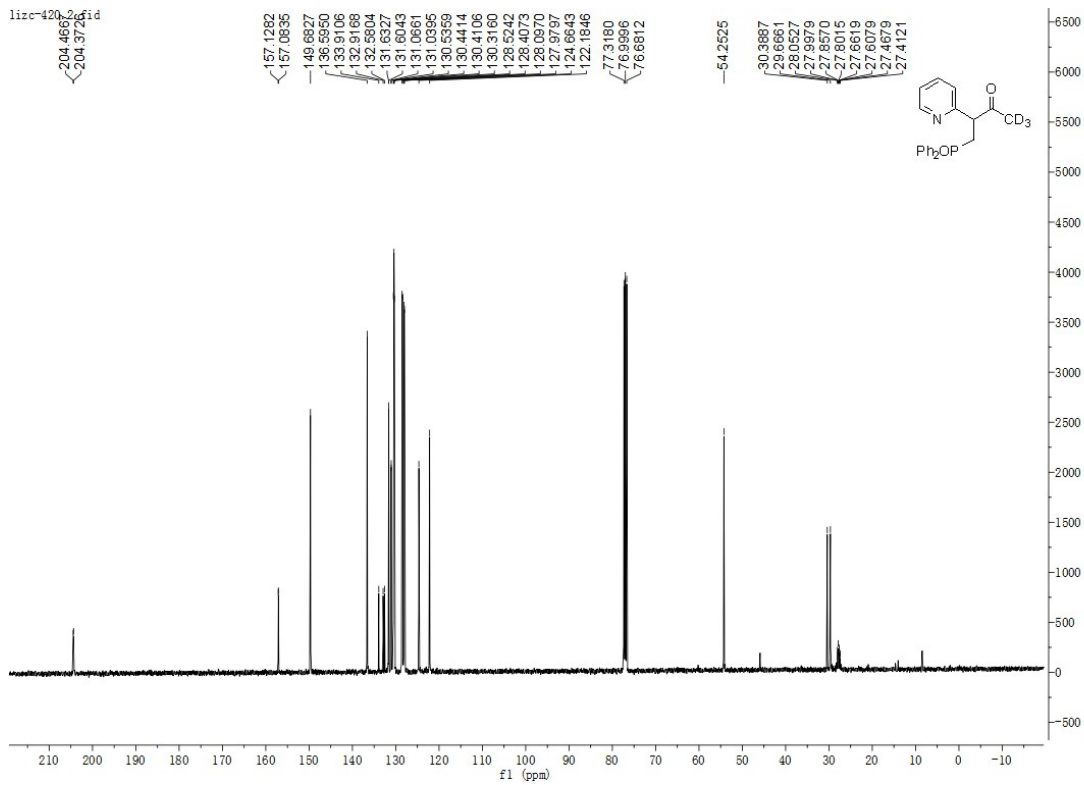
<sup>1</sup>H NMR

lizc-420.4.fid



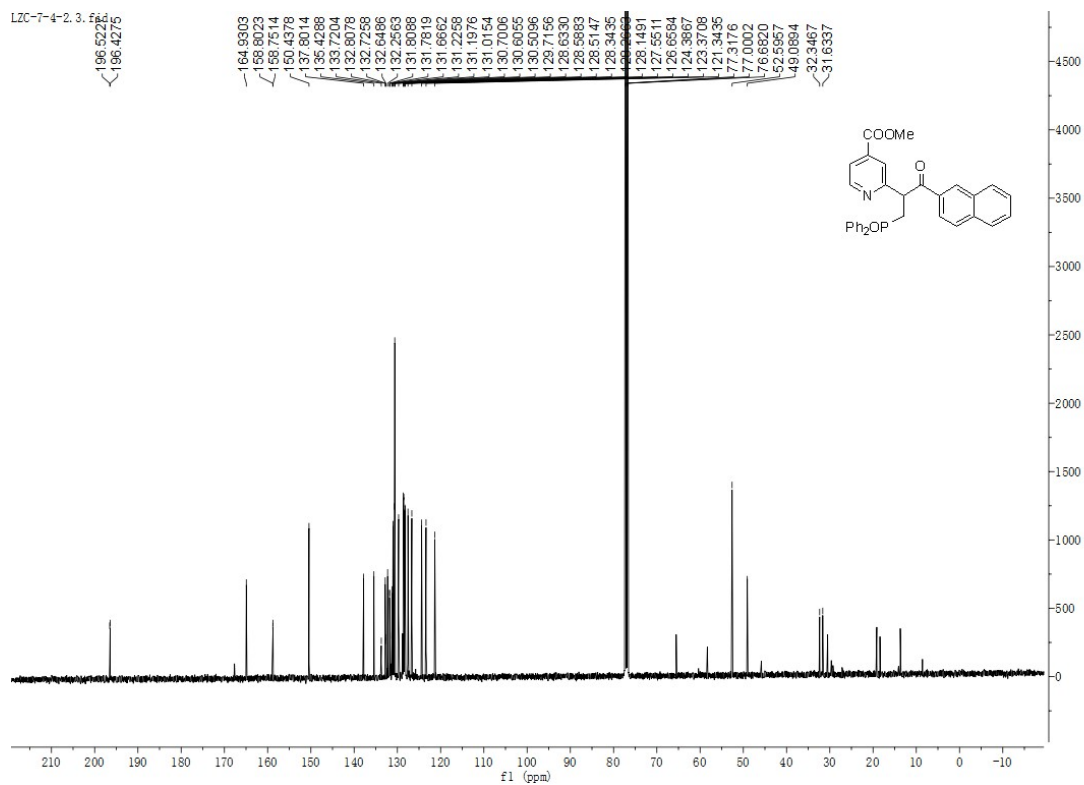
### <sup>13</sup>C NMR

lizc-420.2.fid



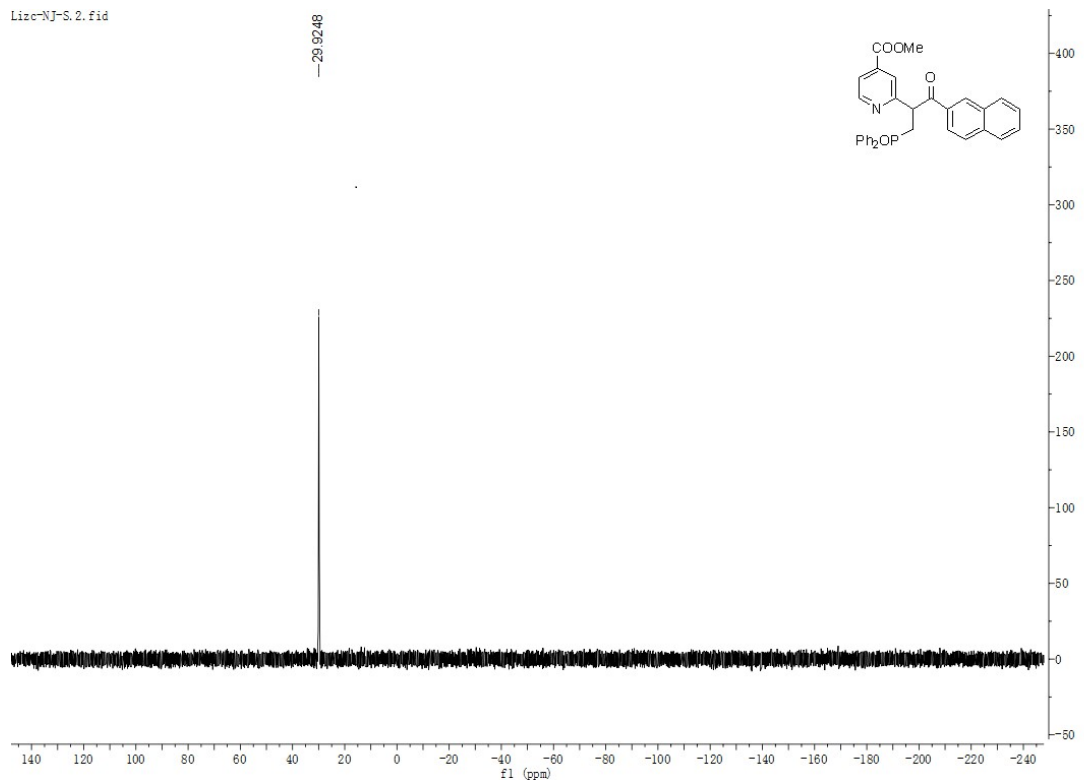
### <sup>31</sup>P NMR





### <sup>31</sup>P NMR

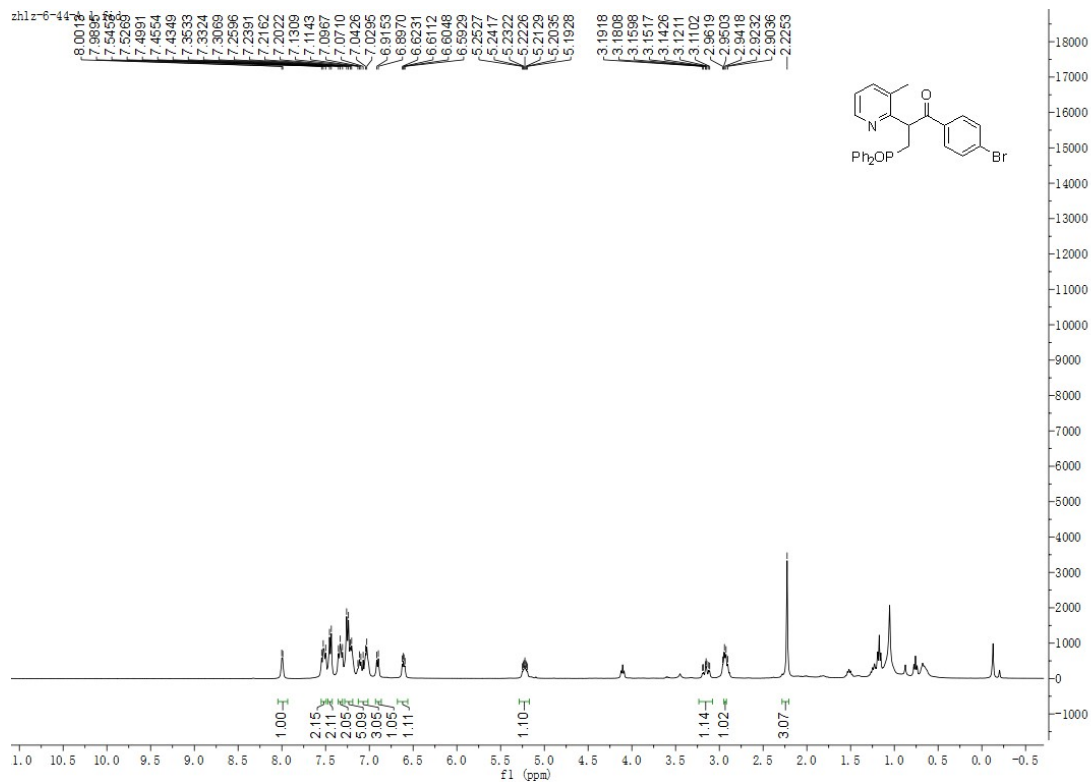
Lizc-NJ-S.2.fid



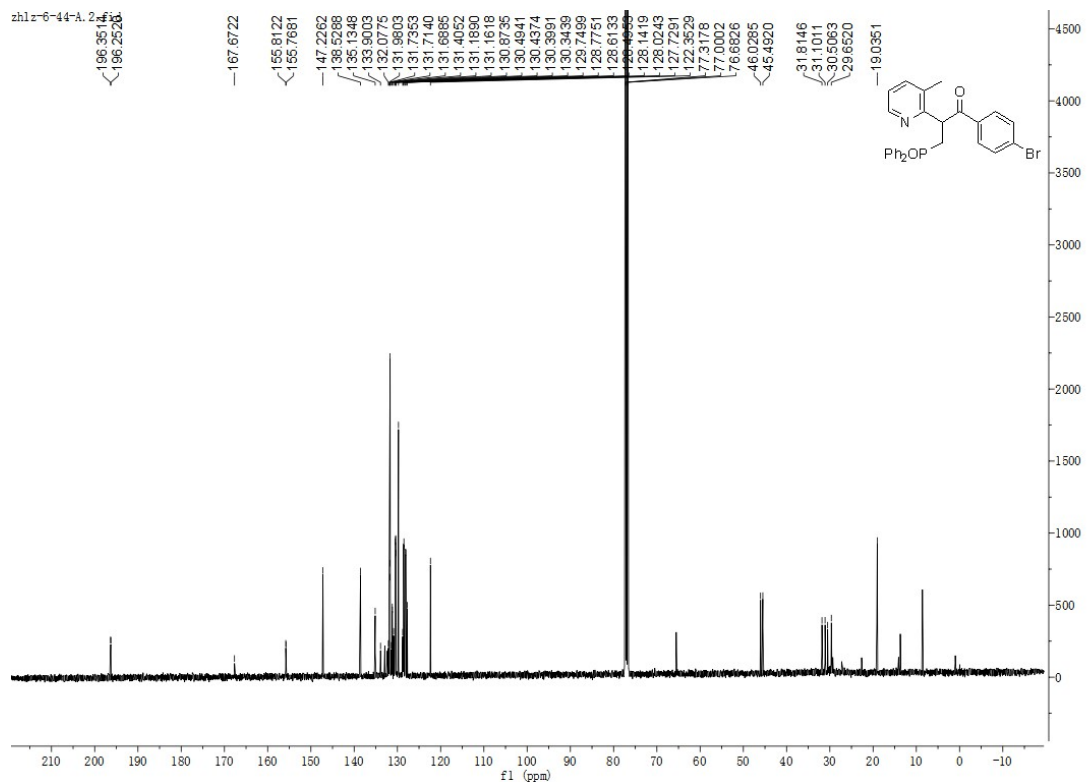
## 26. 1-(4-bromophenyl)-3-(diphenylphosphoryl)-2-(3-methylpyridin-2-yl)propan-1-one (4bb)

<sup>1</sup>H NMR



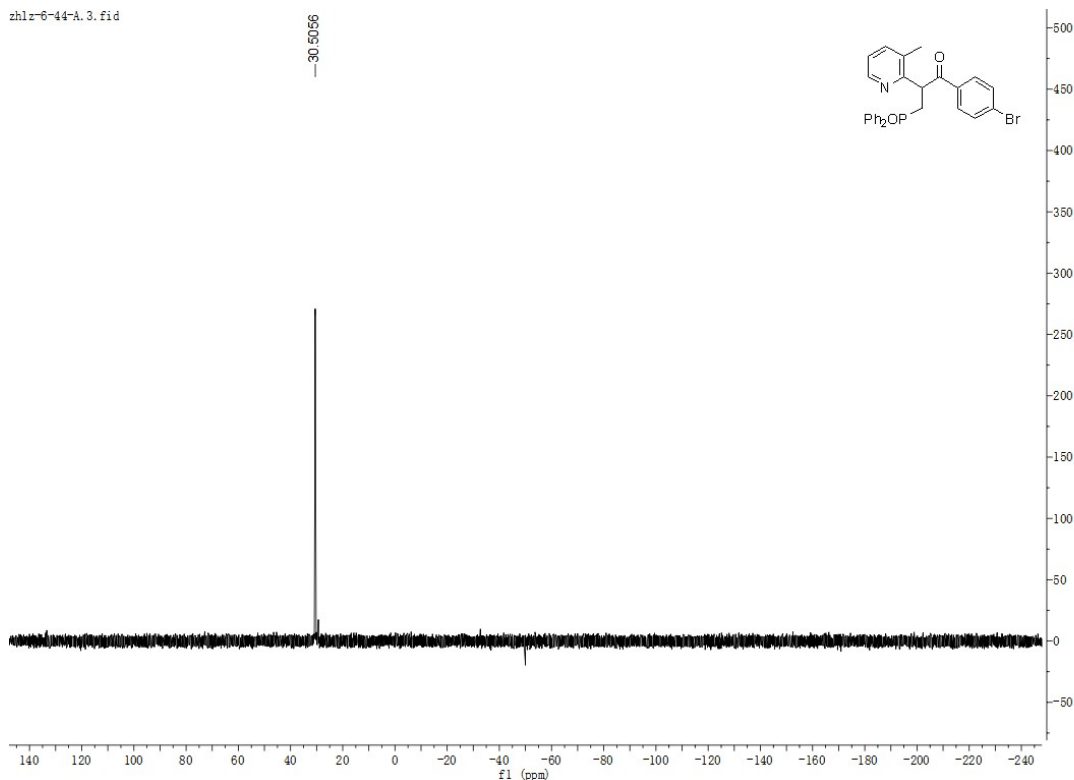


### <sup>13</sup>C NMR



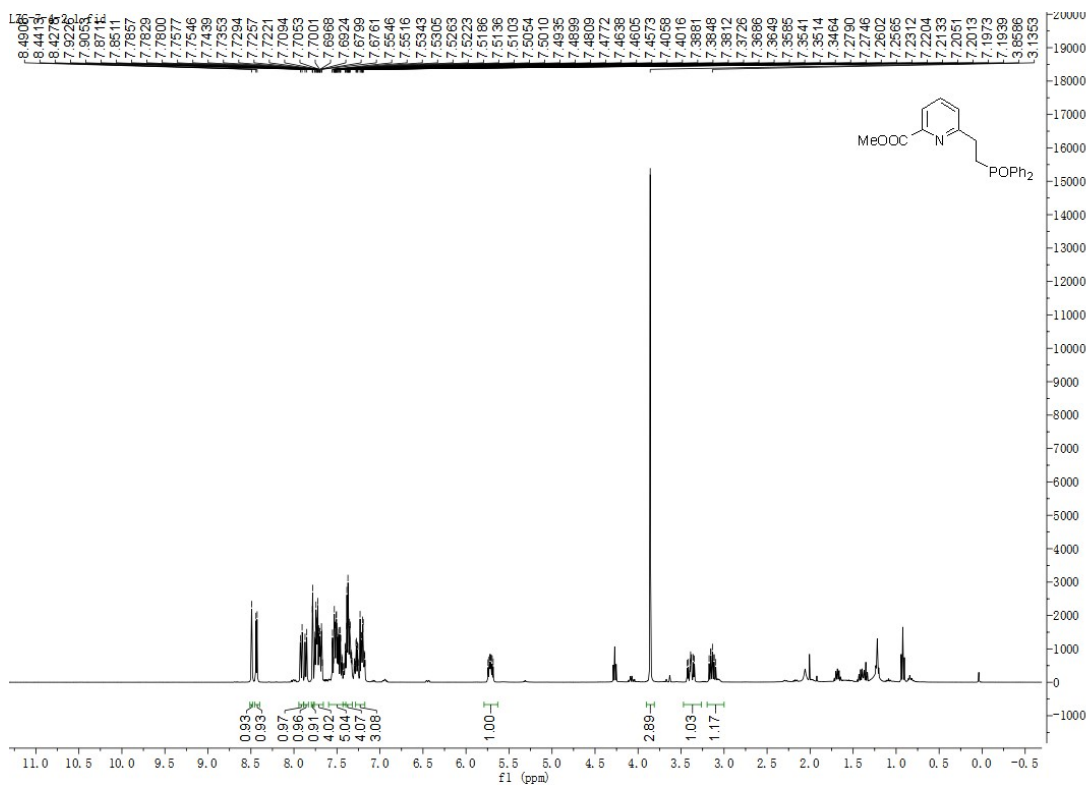
### <sup>31</sup>P NMR

zh1z-6-44-A.3.fid

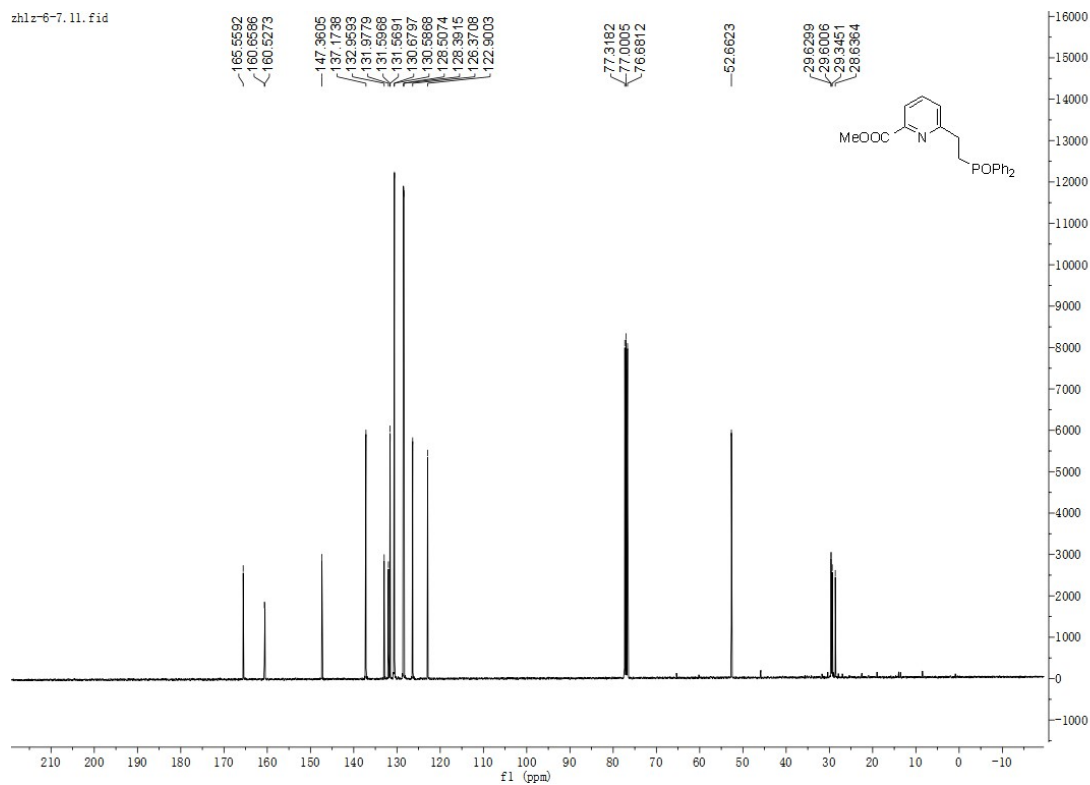


## 27. methyl 6-(2-(diphenylphosphoryl)ethyl)picolinate (4bc)

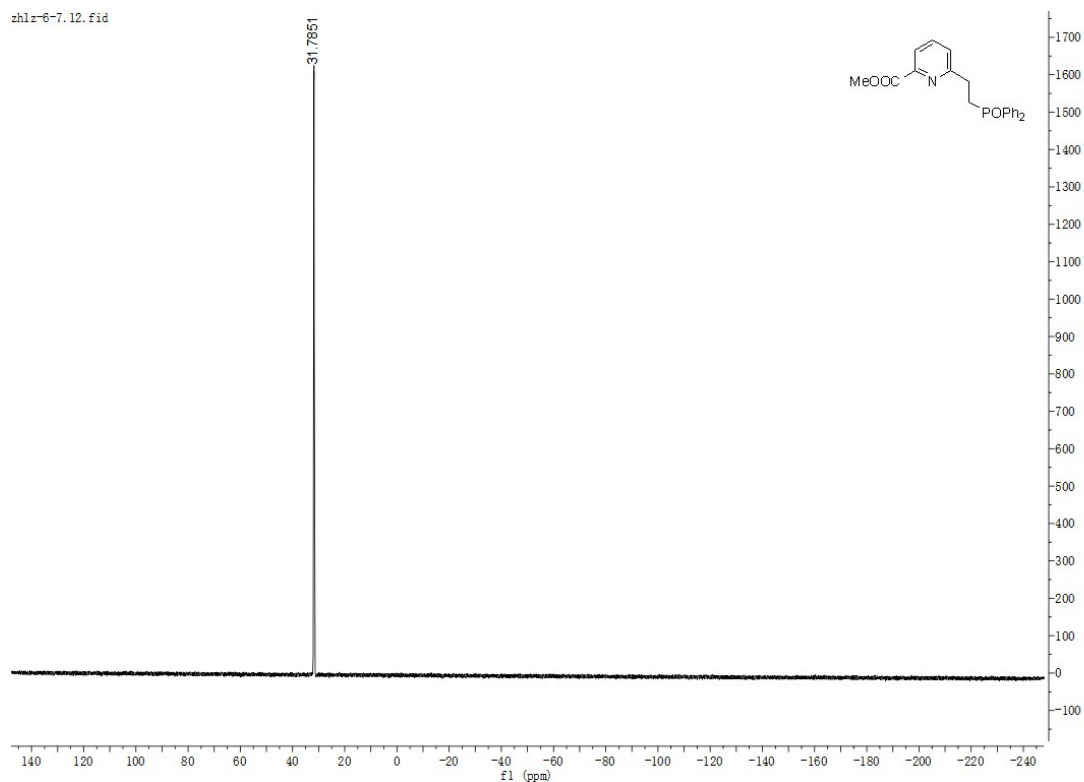
<sup>1</sup>H NMR



<sup>13</sup>C NMR



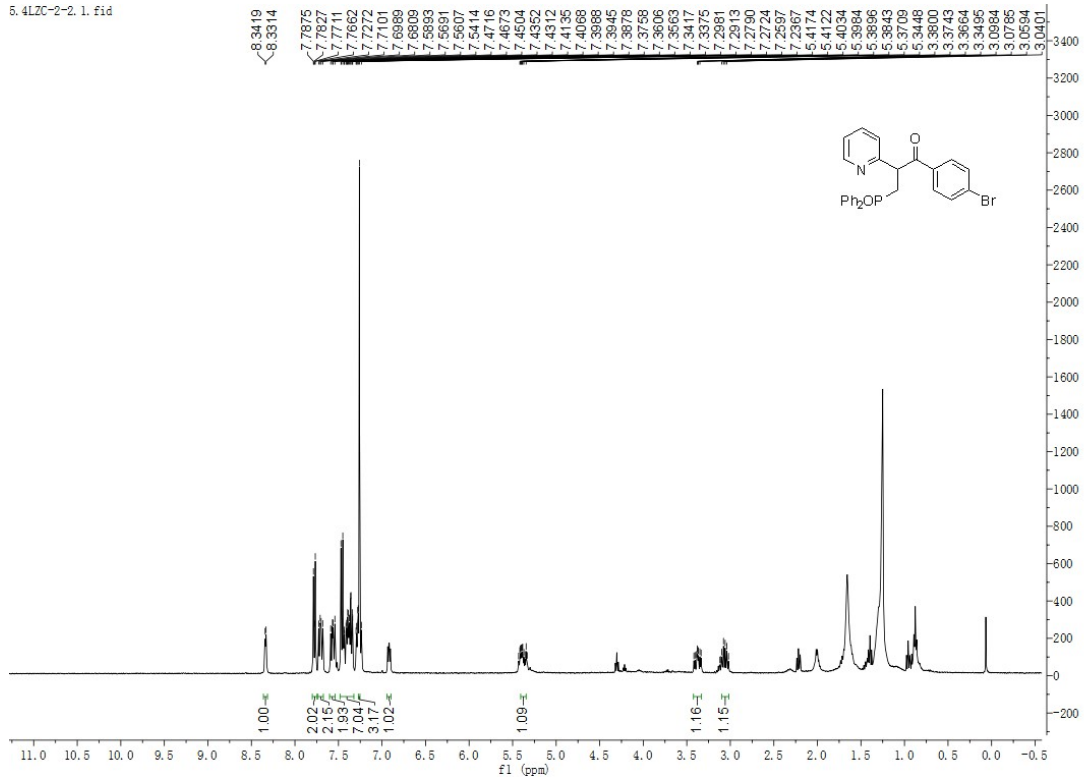
### <sup>31</sup>P NMR



## 28. 1-(4-bromophenyl)-3-(diphenylphosphoryl)-2-(pyridin-2-yl)propan-1-one (4bd)

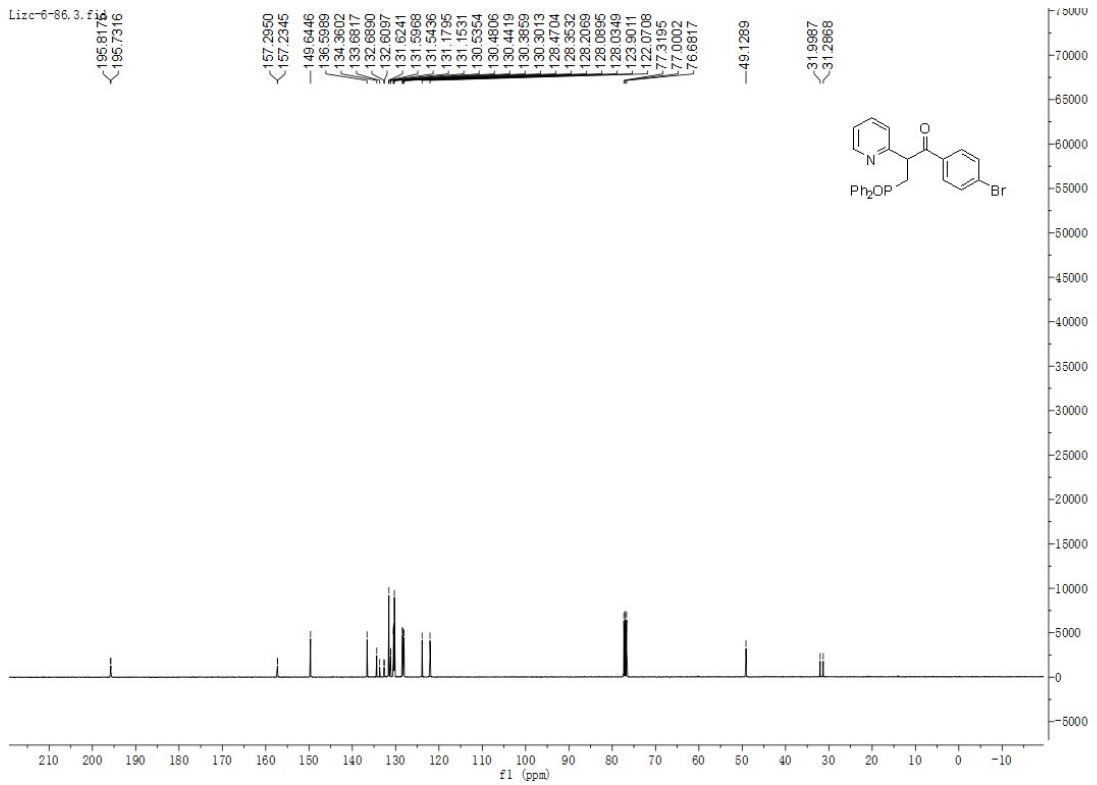
### <sup>1</sup>H NMR

5. 4LZC-2-2. 1. fid

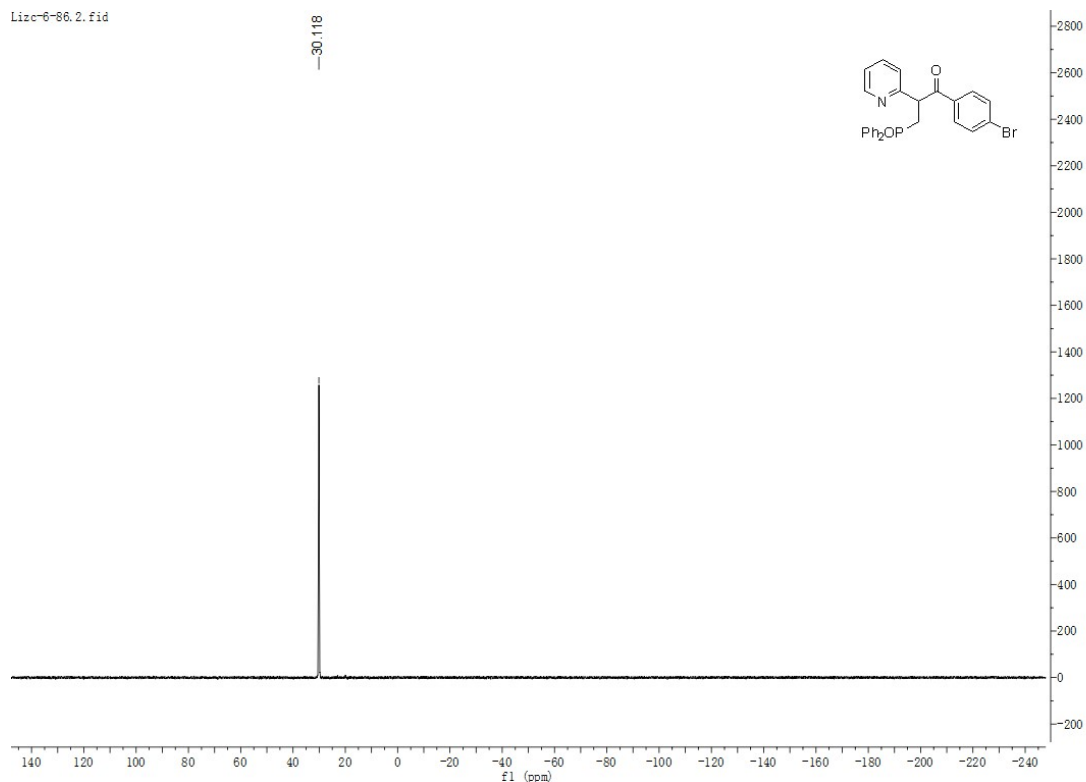


### <sup>13</sup>C NMR

Lizc-6-86.3.fid

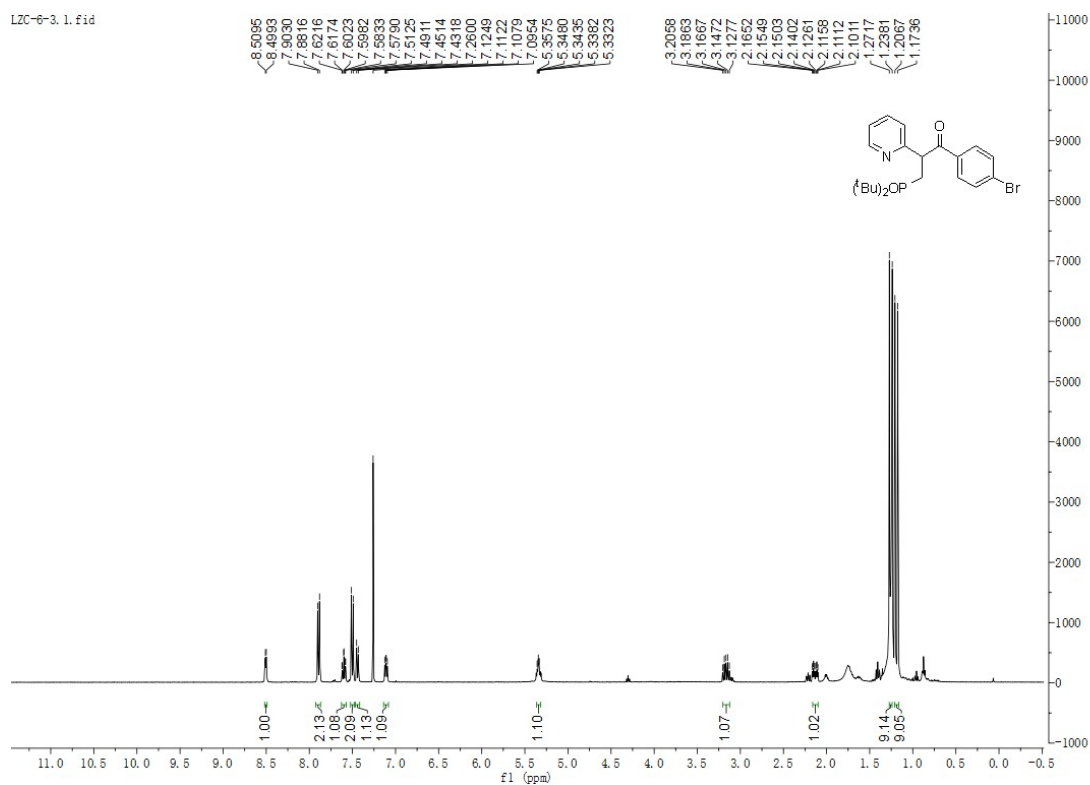


### <sup>31</sup>P NMR

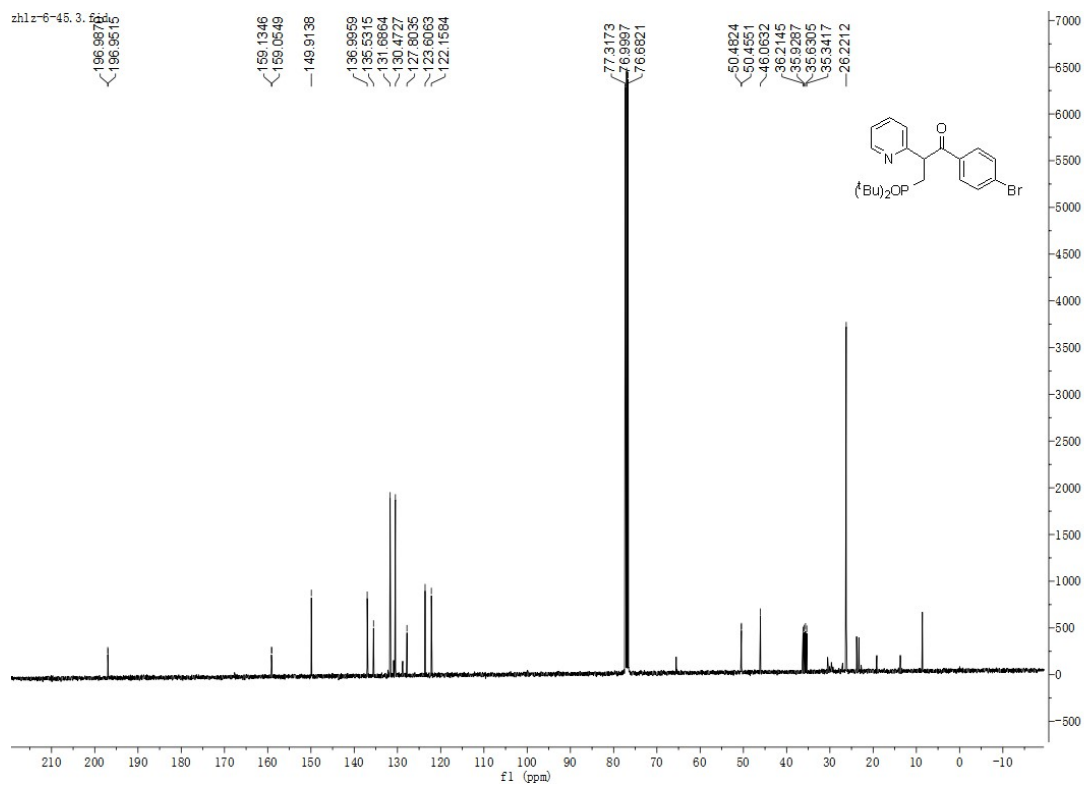


## 29. 1-(4-bromophenyl)-3-(di-tert-butylphosphoryl)-2-(pyridin-2-yl)propan-1-one (4be)

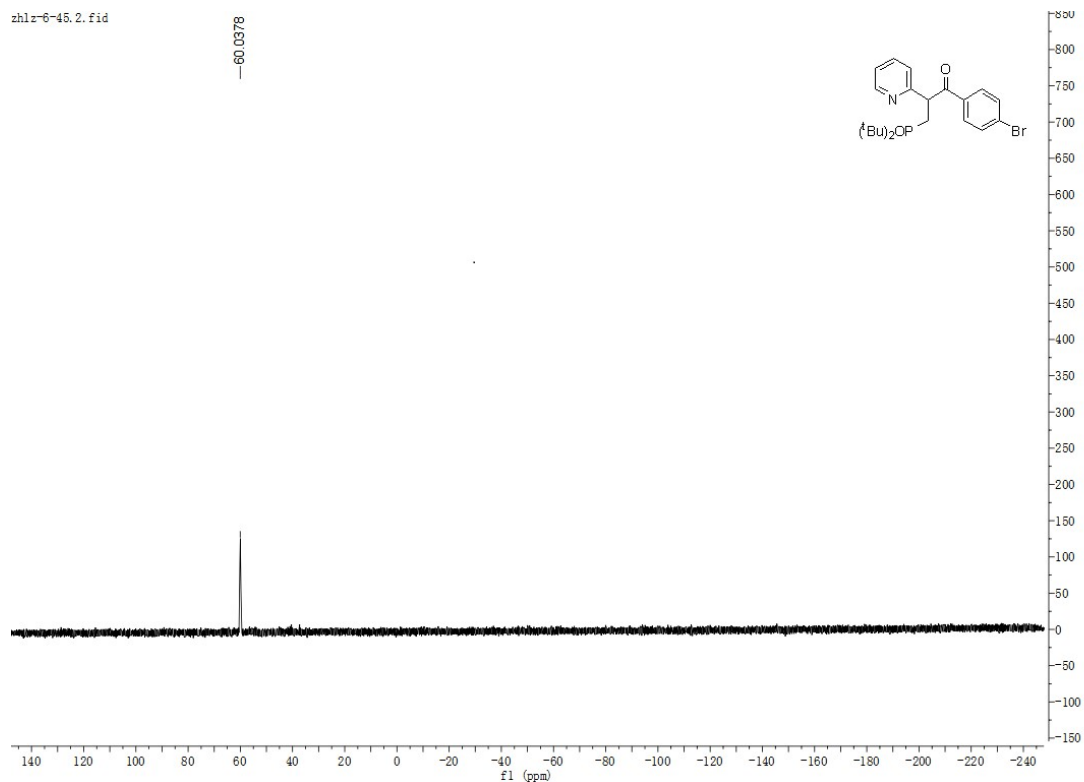
<sup>1</sup>H NMR



<sup>13</sup>C NMR



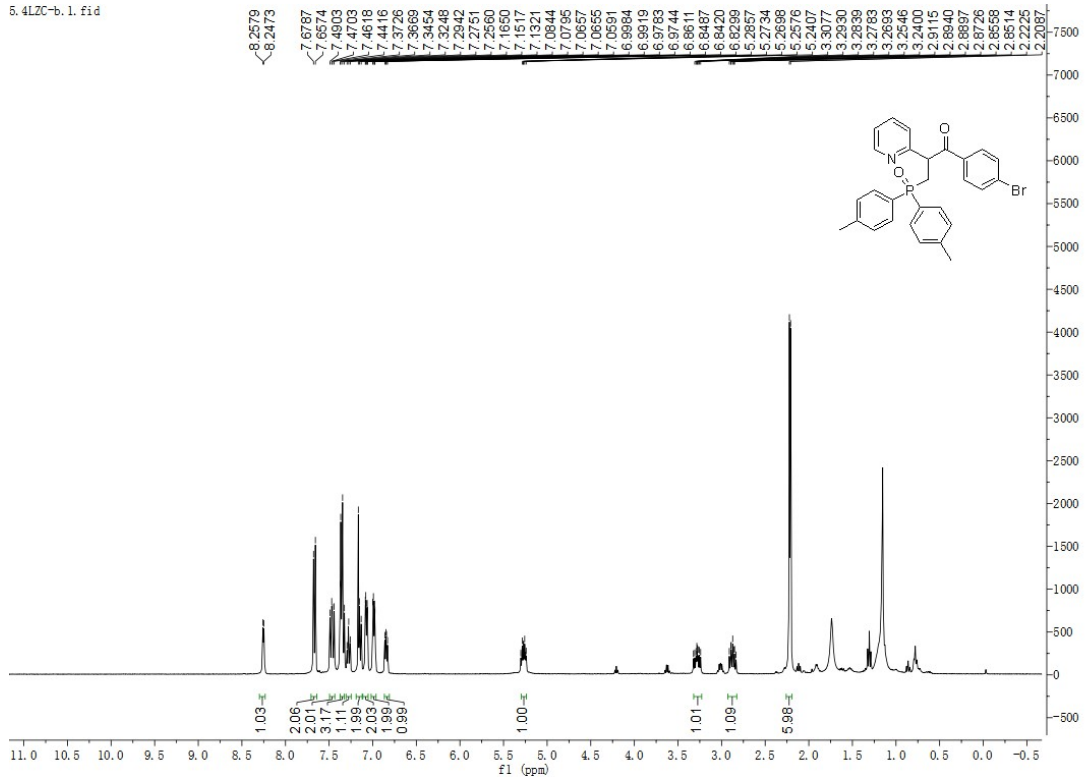
### <sup>31</sup>P NMR



## 30. 1-(4-bromophenyl)-3-(di-p-tolylphosphoryl)-2-(pyridin-2-yl)propan-1-one (4bf)

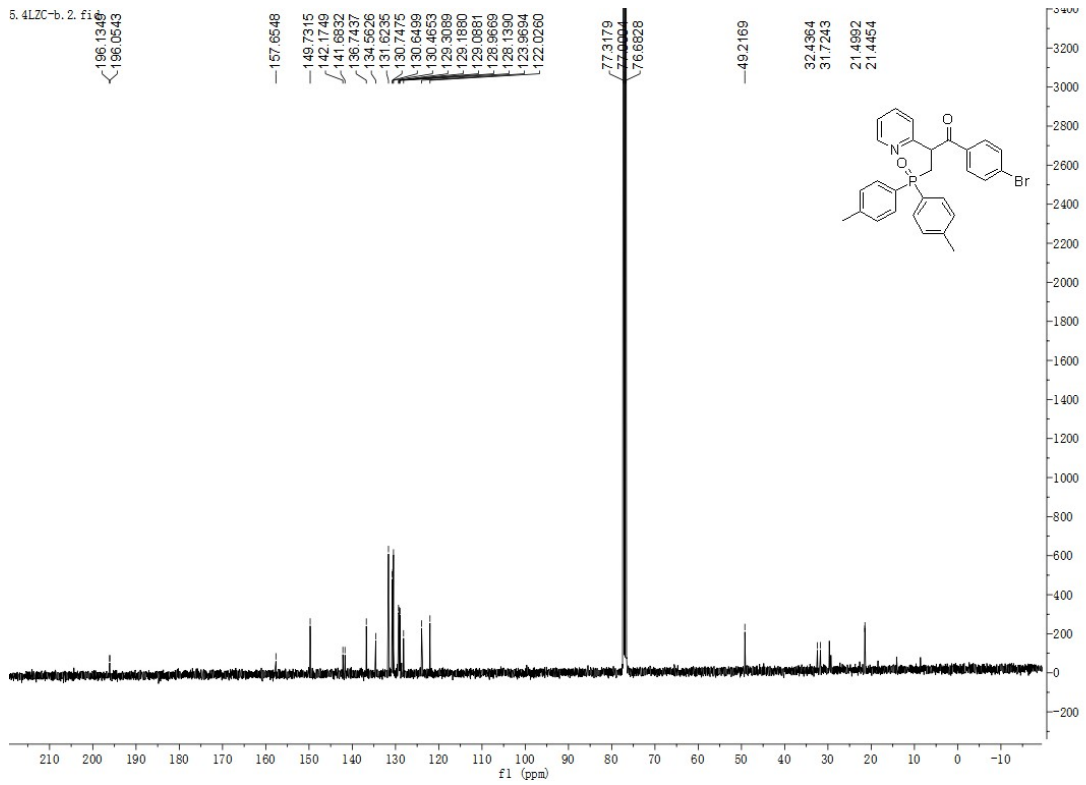
### <sup>1</sup>H NMR

5. 4LZC-b. 1. fid



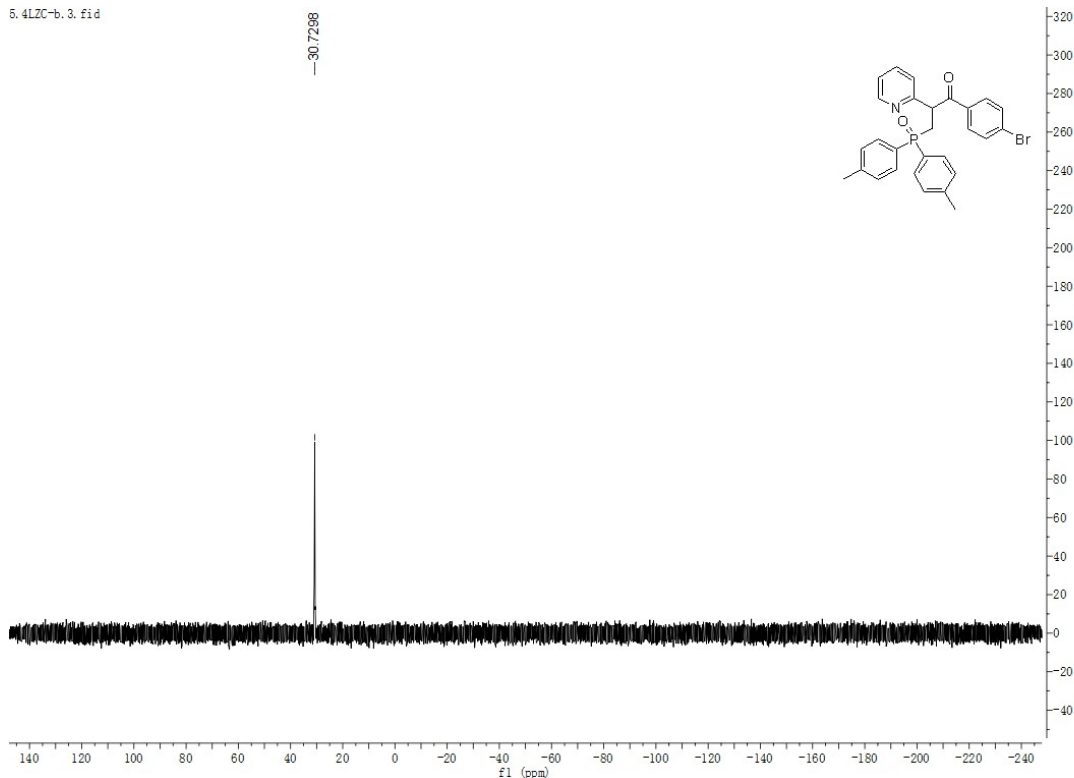
### <sup>13</sup>C NMR

5. 4LZC-b. 2. fid



### <sup>31</sup>P NMR

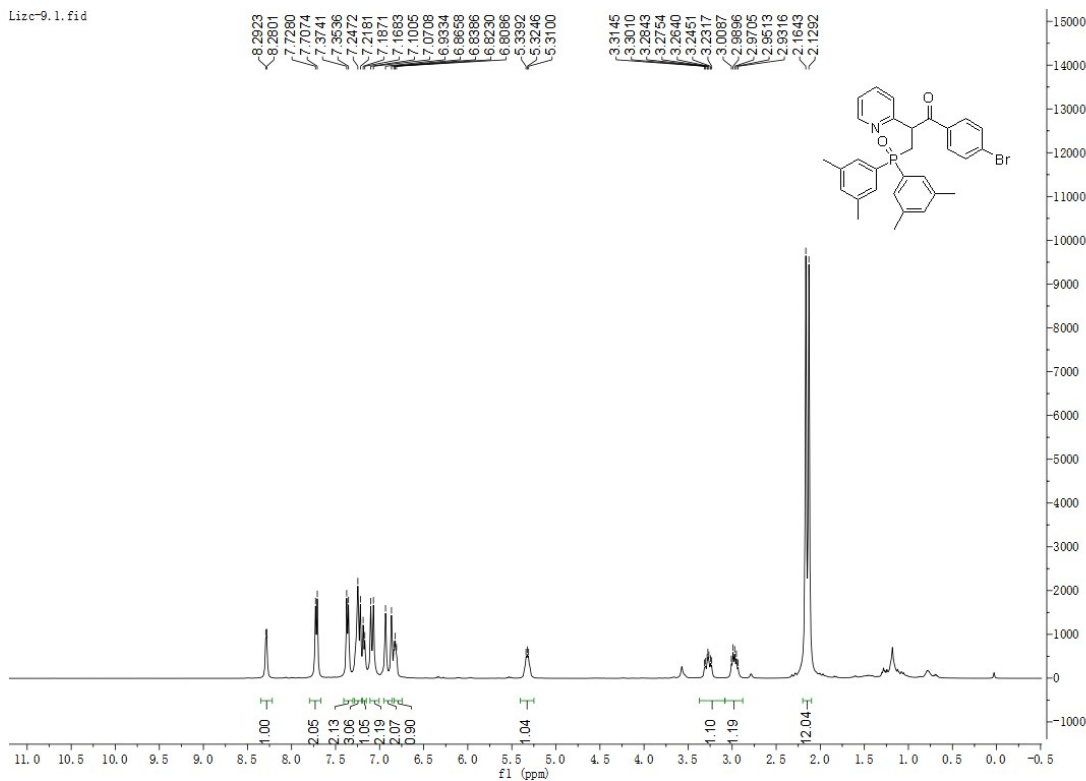
5.4LZC-b. 3. fid



### 31. 3-(bis(3,5-dimethylphenyl)phosphoryl)-1-(4-bromophenyl)-2-(pyridin-2-yl)propan-1-one(4bg)

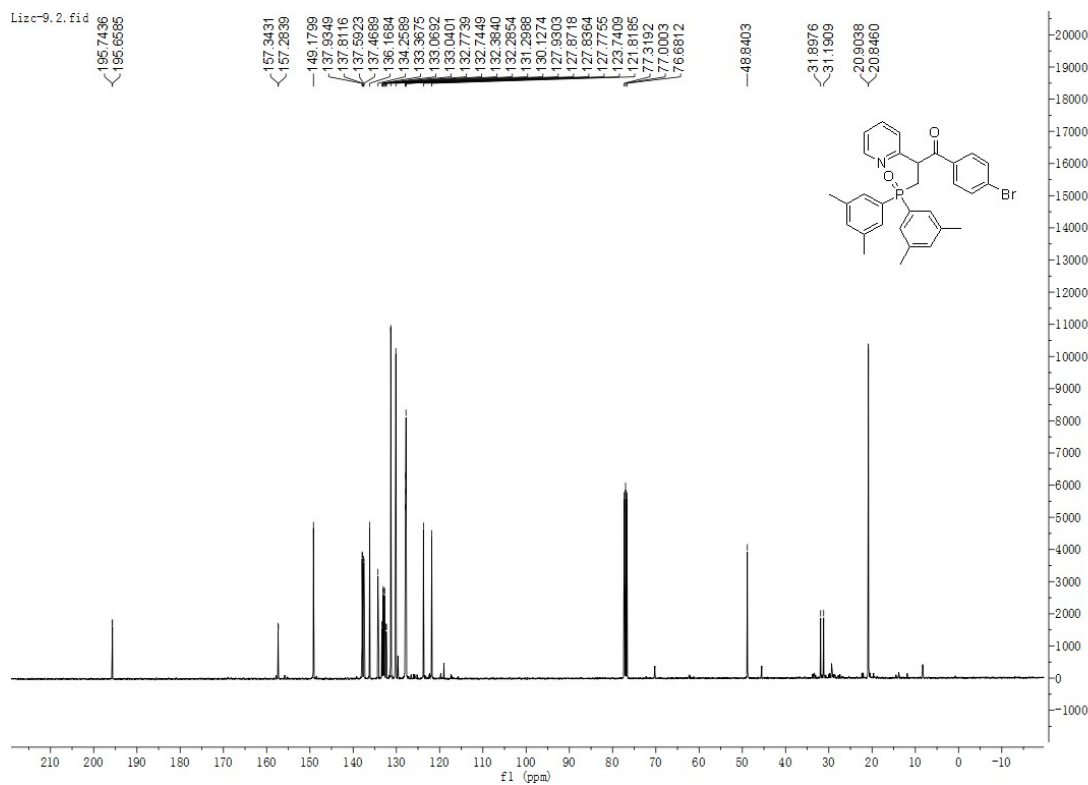
<sup>1</sup>H NMR

Lize-9.1. fid

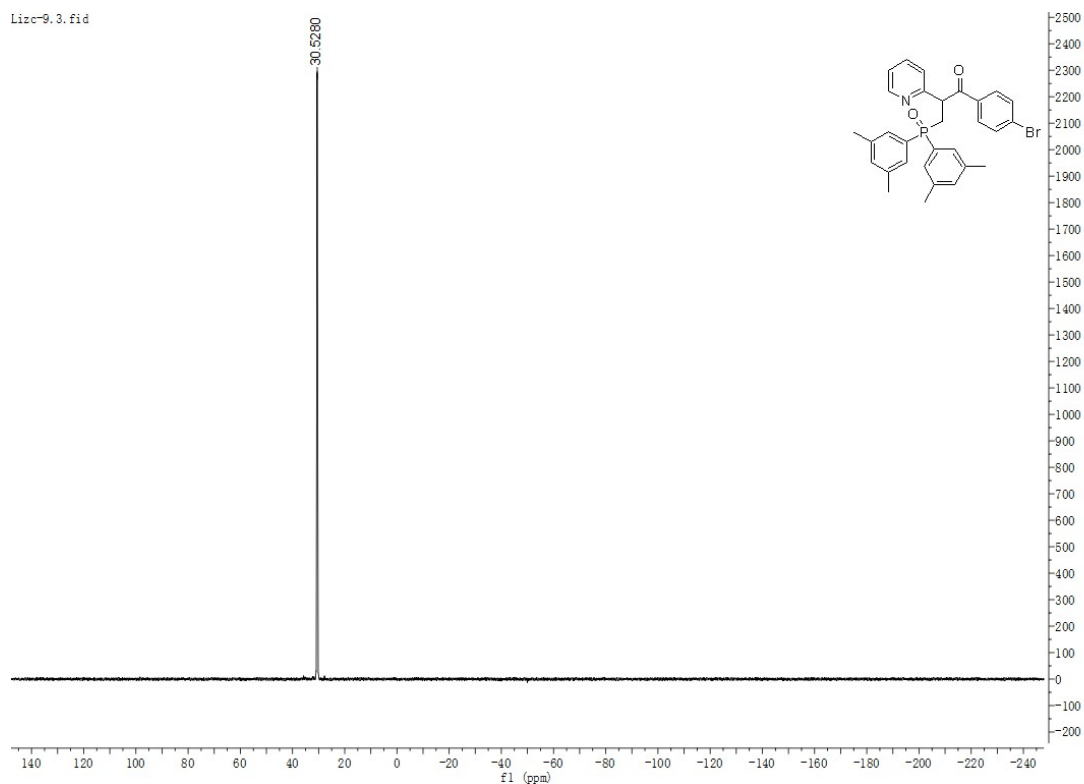


<sup>13</sup>C NMR





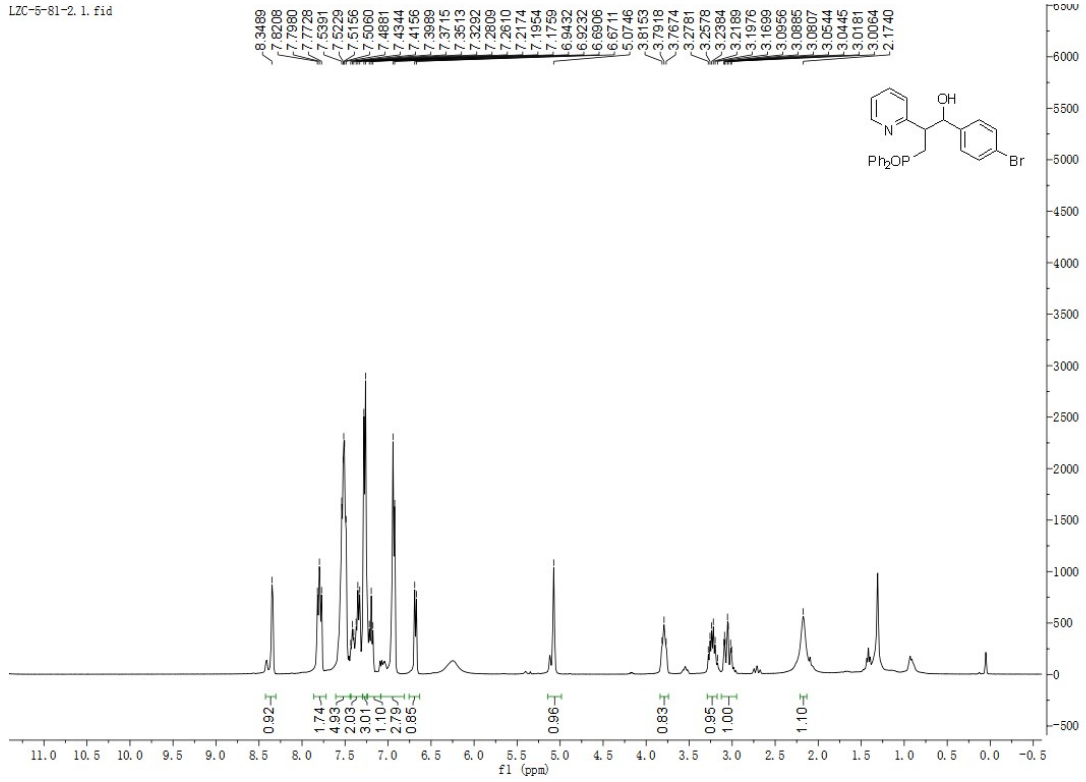
### <sup>31</sup>P NMR



## 32. (3-(4-bromophenyl)-3-hydroxy-2-(pyridin-2-yl)propyl)diphenylphosphine oxide (4ac-1)

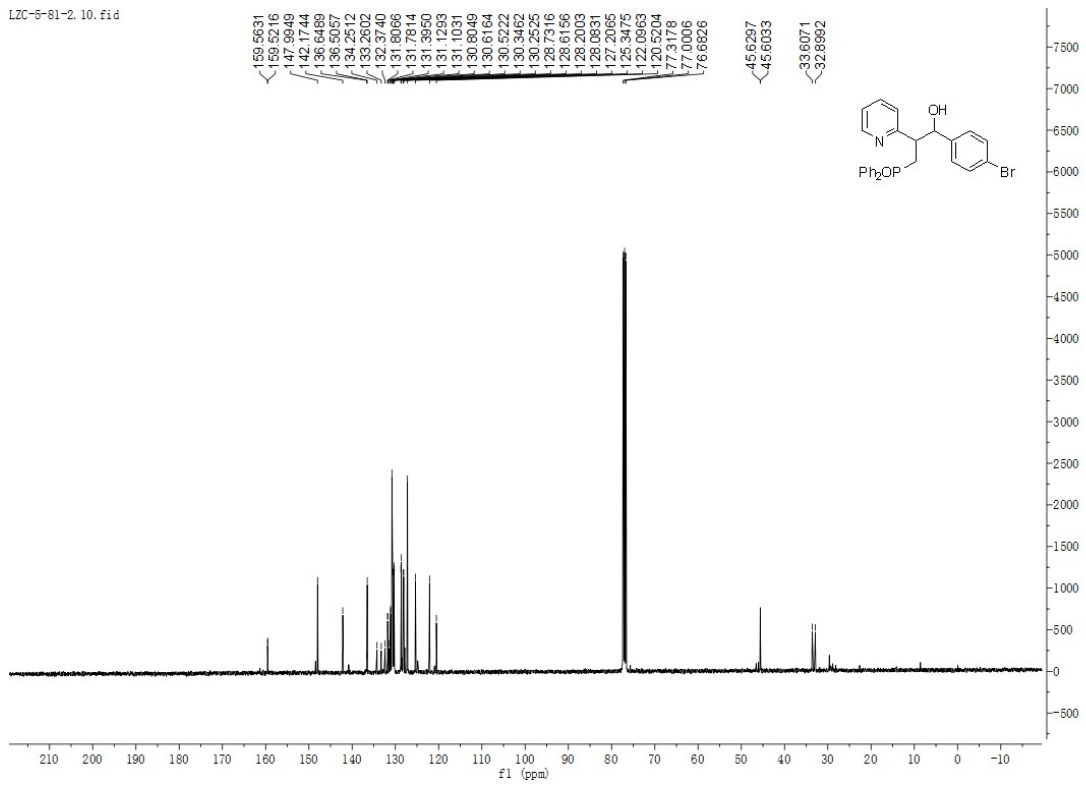
<sup>1</sup>H NMR

LZC-5-81-2.1.fid



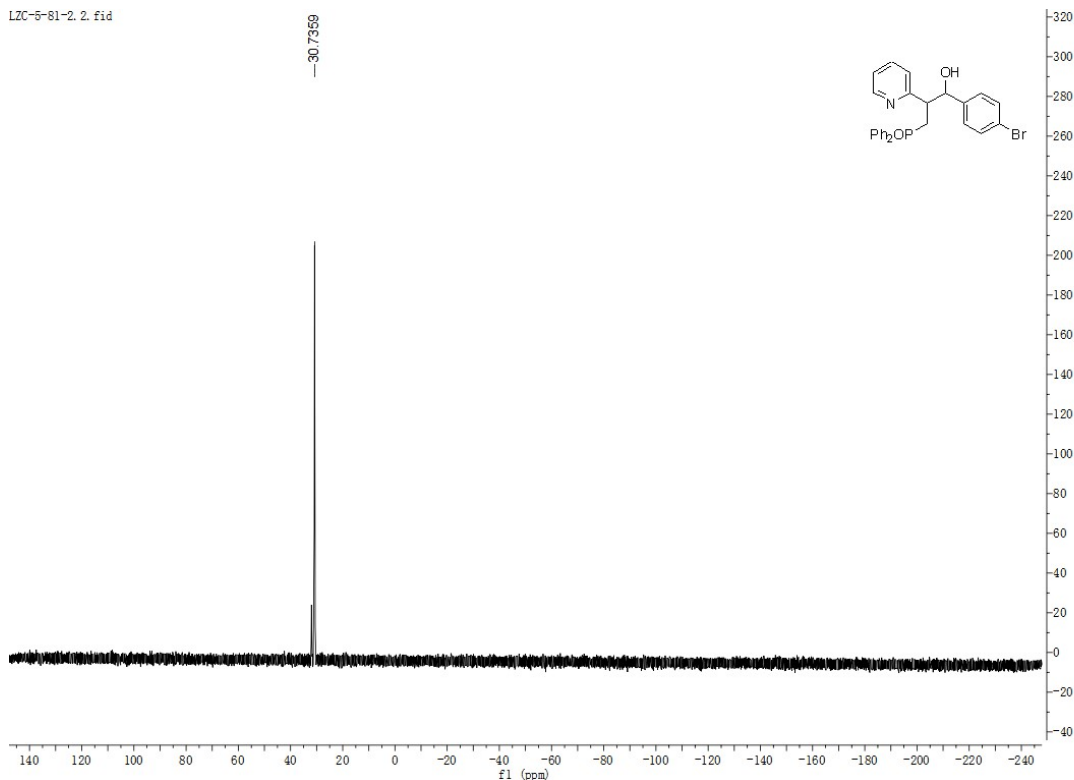
### <sup>13</sup>C NMR

LZC-5-81-2.10.fid



### <sup>31</sup>P NMR

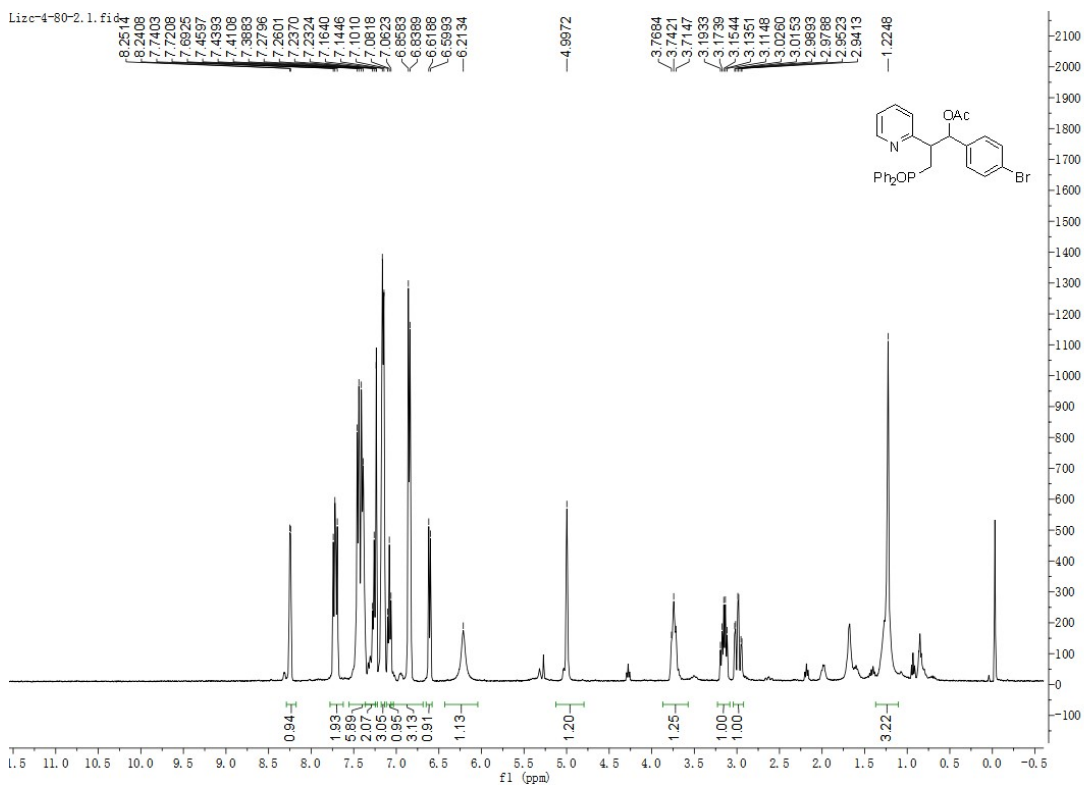
LZC-5-81-2.2.fid



### 33. 1-(diphenylphosphoryl)-2-(pyridin-2-yl)hept-6-en-3-one (4ac-2)

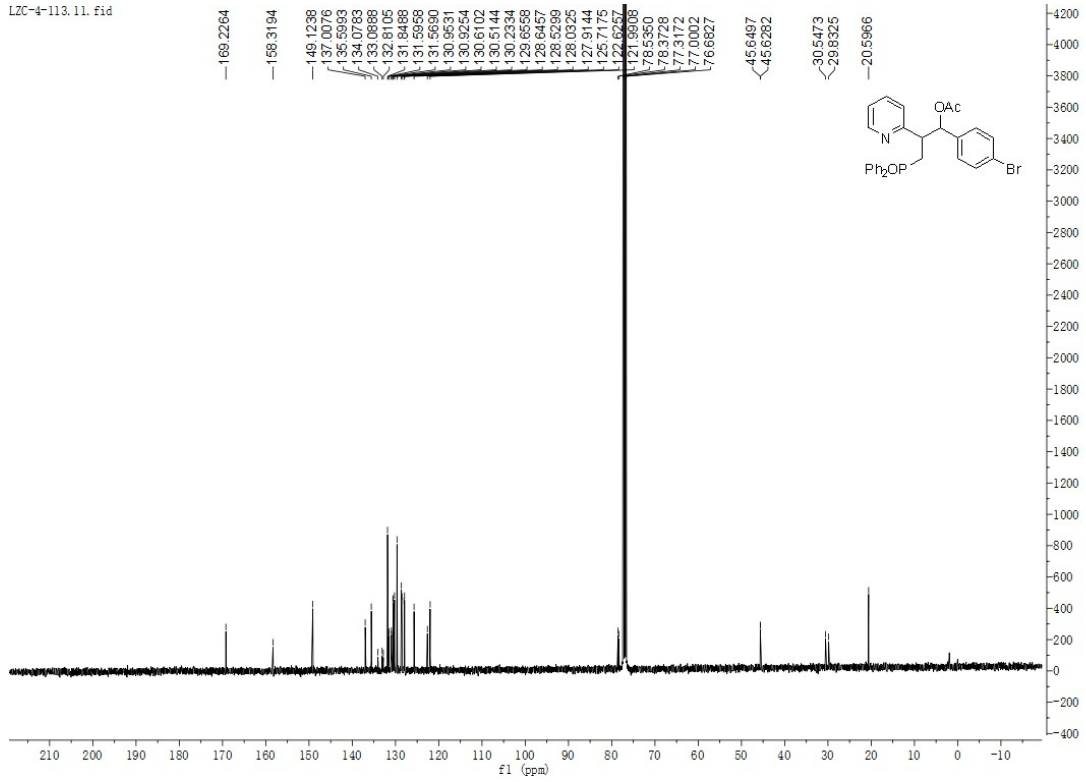
<sup>1</sup>H NMR

Lizc-4-80-2.1.fid



<sup>13</sup>C NMR

LZC-4-113.11.fid



### <sup>31</sup>P NMR

Lizc-4-80-2.3.fid

